

MICROWAVE FIRING OF LOW-PURITY ALUMINA

By

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Microwave (hybrid) firing (MHF) offers many potential benefits to ceramic processing, including reductions of both the production time and temperature required to fire ceramic components. These benefits result from features of microwave processing, such as volumetric heating and enhanced diffusion within the ceramic body. Further investigation into issues such as scale-up, microwave susceptors, and mechanical properties of MHF samples is needed if this technique is to gain acceptance by industry.

An experimental study was undertaken to investigate MHF as an alternative to conventional electric or gas firing for the production of small, uniform batches of alumina samples. A MHF assembly was designed and developed so that comparisons could be made between the densification and mechanical properties (hardness, strength, and fracture toughness) of samples fired using the two techniques. In addition to these comparisons, the effect of alumina purity on MHF and the effect of energy partitioning on sample densification were also evaluated.

Through this study, it was determined that MHF was a viable alternative to conventional firing for the production of uniform batches of low-purity alumina pieces. The firing temperature required to reach identical levels of densification in the alumina samples was from 75 to 200°C lower for MHF as compared to conventional firing.

Possible reasons for the enhanced densification of MHF relative to conventional firing include the microwave volumetric heating phenomenon and a non-thermal effect of microwaves on the viscosity and/or surface energy of the glassy phase present in the low-purity alumina.

Some control of the sample densification rate was afforded through control of the energy partitioning between microwave volumetric heating and conventional surface heating. It was found that increasing the ratio of conventional surface heating to microwave volumetric heating could decrease the sample densification rate.

The mechanical properties of samples fired to identical relative densities (95% relative density) using the two techniques were statistically similar. Hardness was uniform in samples processed using either technique.

CHAPTER 1 INTRODUCTION

There is a continual need for research in order to improve the fabrication process of ceramics, thereby producing mechanically improved parts, or lowering processing costs. The focus of the current study is to objectively evaluate an alternative to traditional techniques used for sintering of the ceramic body.

The alternative firing technique combined surface heating with direct volumetric heating of ceramics through application of both traditional radiant heating techniques and an applied microwave field. This technique has been termed microwave-assisted or microwave hybrid heating.

Studies on the utilization of microwaves for sintering of ceramic materials have shown that microwave sintering offers advantages over conventional sintering techniques. A study by Janney [Jann91] found that the apparent activation energy for microwave sintering of alumina at 28 GHz was significantly lower than that for conventional firing (160 kJ/mol vs. 575 kJ/mol). This decrease in the apparent activation energy translated into a 300 to 400°C reduction in the required sintering temperature for microwave sintering compared to that required for conventional sintering.

In addition to reducing the activation energy and required sintering temperature, microwave firing, when assisted by either gas or electric furnace firing, can provide large energy savings over purely conventional techniques or microwave firing alone [Figure 1-

1]. From the figure, it is evident that microwave-assisted heating can reduce the relative energy costs from 5 to 95% over either gas or electric furnace, or microwave firing alone.

In the current study, the microwave hybrid heating technique is not assisted by either gas-firing or electric-firing. Instead, the infrared energy transfer component is supplied by microwave absorbing materials (susceptors). These susceptors partially absorb incident microwave energy and reradiate it in the form of infrared energy. This radiant energy then heats the surface of the sample.

The combined mechanisms of energy transfer found in microwave hybrid heating serve to equilibrate the temperature profile within the ceramic body. The radiant heating from susceptors has the additional effect of preheating ceramics to temperatures where direct microwave heating is possible.

The merits of microwave hybrid heating has been evaluated against conventional furnace firing based on the densification and resulting mechanical properties of Coors AD998 and Coors AD85 alumina bodies. Both grades of alumina have been used in armor applications as well as in other technical areas. Coors AD998 is a relatively pure alumina (~99.8%) and is densified through solid-state sintering mechanisms. Coors AD85 alumina is less pure than Coors AD998 alumina, and utilizes a glassy liquid phase to assist the sintering process. Because of this utilization of liquid phase sintering, Coors AD85 requires a lower firing temperature than the higher purity alumina, provided all other factors are similar.

The hybrid part of microwave hybrid heating was supplied by a susceptor composed of an alumina cement matrix and a microwave absorbing phase of silicon carbide particles. A major focus of this study was to design and develop the susceptors

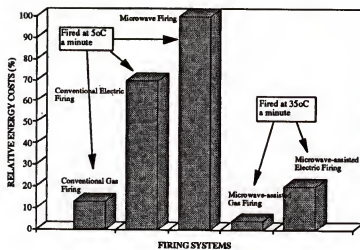


Figure 1-1. Relative Energy Costs for the Firing of Alumina Cylinders [Wroe93].

for scale-up to batch processing (Chapter 3). The objective was to scale-up from one sample (12 to 15 grams) per batch to more than ten samples per batch.

Production of larger batches of samples was crucial to this study and to the further development of this technique for industrial use. This study relied on batch processing to produce adequate quantities of samples for statistical assessment of sample strength. Economical industrial implementation of this technique would likely require continuous processing of larger batches of samples. The susceptor and insulation development found in this study could provide insight into the factors influencing successful production of these larger batches.

Additional study on the susceptors was undertaken to evaluate the effect of increased weight fractions microwave absorbing phase in the susceptors on sample densification and selected mechanical properties (hardness and strength). Several different ratios of absorbing phase (SiC) to matrix phase (alumina) were studied in order to evaluate this effect. Specifically, susceptors with 10, 20, 30, and 60 weight percent absorbing phase were investigated. The study of this ratio and comparisons to conventional firing were conducted at several different firing temperatures in order to develop firing schedules that resulted in nearly fully dense bodies.

Once fully dense bodies were produced, hardness testing and indented strength testing on the microwave hybrid heated and conventionally fired samples were initiated. To assess the uniformity of samples produced by the different firing techniques, hardness testing was conducted across the surface and cross-sections of the samples. Based on the results of these mechanical and densification studies, a recommendation is made for

further study of microwave hybrid heating as an alternative firing technique for the production of alumina ceramics.

The objectives of the study are to:

- develop a reusable and reliable system for microwave hybrid-firing which uniformly sinters all batch processed samples (10 + samples per batch) to relative densities of at least 95%
- evaluate the firing characteristics and performance of microwave hybrid-fired alumina samples relative to conventionally processed samples
- evaluate the effect on sample densification of the relative amounts of microwave energy to radiant energy (susceptors) that are used to densify the samples
- evaluate the effect of microwave hybrid-firing on the sintering and performance of alumina bodies that densify through solid-state sintering mechanisms (high purity), and those that densify through liquid-phase sintering mechanisms (low purity).
- based on the results of the study and knowledge of sintering mechanisms, provide explanations for differences between the firing characteristics and performance of microwave hybrid-fired alumina samples relative to conventionally processed samples

The study is organized into several chapters including a literature review, a discussion of susceptor development, a summary of the materials and experimental methods used, the experimental results, a discussion of the results, and the conclusions of the study. An appendix is also included to provide insight into factors that influence ballistic performance of thin ceramics armor tile. A good understanding of these factors is needed should microwave hybrid heating be utilized to fabricate alumina armor tile.

The study begins with a review of literature on the sintering process and microwave heating of ceramics.

CHAPTER 2 LITERATURE REVIEW

Conventional Fabrication of Ceramics

Ceramics are normally fabricated through the densification of particulate compacts at high temperature. A typical fabrication procedure is provided in Figure 2-1. Fine ceramic powders are weighed and poured into a die having the final shape of the part. The particles are compacted together through application of high pressure, resulting in the green ceramic body. Any binders that have been added are then removed at low temperatures (300-600°C). After binder burnout, the ceramic compact is transferred to a kiln or furnace where it is ramped to high temperature (1300+°C) and allowed to soak for a time, and then cooled back to room temperature. During this final heating process, the ceramic is sintered to form a densified body. This sintering process involves both the bonding of adjacent particles and the removal of pores between the starting particles. It is accompanied by the shrinkage of the component [Rich92]. After cooling back to room temperature, any required polishing or rounding is done on the ceramic piece to remove surface flaws that have resulted from processing.

Solid-State Sintering

There are five mechanisms for the mass transport that are possible during the solid-state sintering process. These can be readily viewed using a two-sphere model [Figure 2-2]. Each of the two spheres represents an idealized particle contained within the compact. The mechanisms available for mass transport are evaporation-condensation

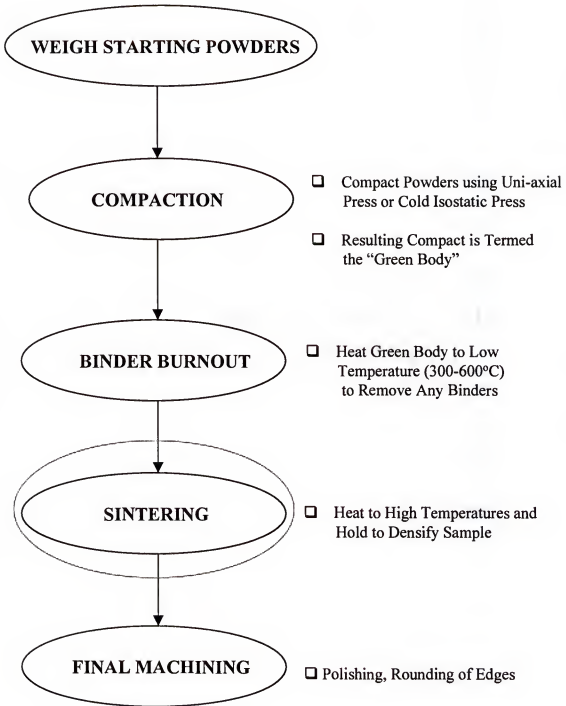


Figure 2-1. Typical Steps for Fabricating Ceramics

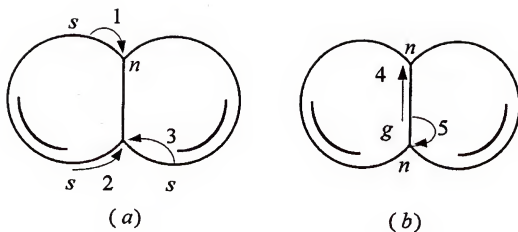


Figure 2-2. Basic Atomic Mechanisms that can Lead to (a) Coarsening and Change in Pore Shape and (b) Denatification [Bars97]. (1) Evaporation-Condensation, (2) Surface Diffusion, (3) Volume Diffusion (Surface to Neck Area), (4) Grain Boundary Diffusion, (5) Volume Diffusion (Grain Boundary to Neck Area).

(path 1), surface diffusion (path 2), volume diffusion (paths 3 and 5), grain boundary diffusion to the neck area (path 4), and viscous/creep flow. Volume diffusion has two sub-cases including volume diffusion from the surface to the neck area (path 3), and volume diffusion from the grain boundary area to the neck area (path 5). All five mechanisms are in competition during the sintering process. Depending on which mechanism dominates, either coarsening or densification of the compact will occur.

Evaporation-condensation, surface diffusion, and lattice diffusion from the surface to the neck area all result in coarsening. These mechanisms do not allow for shrinkage of the compact, and therefore cannot lead to densification. They do lead to a growth in neck size, an increase compact strength, and a change in pore shape. The major driving force for these mechanisms is partial pressure differences caused by local variations in curvature.

Densification occurs only when there is mass transport from the grain boundary region to the neck or pore area. Therefore, the only mechanisms that can lead to densification are grain boundary diffusion, and bulk diffusion from the grain boundary area to the neck area. Both grain boundary diffusion and bulk diffusion involve movement of ions from the grain boundary region to the neck region. The major driving force for these modes of transport are curvature induced vacancy concentrations.

The mechanism that proceeds at the fastest rate determines whether the surface energy will be reduced by coarsening or by densification. Models have been developed to provide a better understanding of the dominance of certain mechanisms over others.

The solid-state sintering process can be modeled as three stages divided on the basis of physical events occurring in the compact. A cartoon of these stages is provided

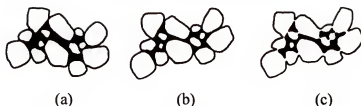
in Figure 2-3. The initial sintering stage is marked by particle rearrangement, neck formation between particles, and grain boundary formation. Rearrangement of the particles occurs as a slight movement or rotation of adjacent particles in order to facilitate an increase in the number of points of contact. Bonding at the points of contact occurs where both the surface energy is highest and material transport is possible [Rich92]. The decrease in porosity during this initial sintering stage is normally less than 12% [Reed95].

The majority of the porosity within the compact is eliminated during the intermediate stage of sintering. Concurrent with this porosity removal is a large amount of sample shrinkage. The shrinkage and pore removal occurs through a combination of neck growth and shrinkage of pores near grain boundaries. The pore phase, however, remains continuous throughout the entire intermediate stage of sintering.

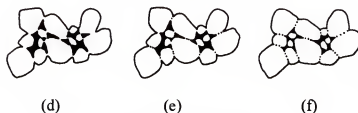
The final stage of sintering is denoted by the formation of closed pores within the compact. These closed pores are gradually eliminated at the grain boundaries. After the majority of grain boundary pores are consumed, grains begin to grow at a much faster rate. These fast growing grains can trap any remaining grain boundary pores within the grain structure. The trapped porosity and any large remaining pores are difficult to remove, and limit the relative density a ceramic can achieve.

Control of grain growth that occurs in the final stage of sintering is very important. Not only can fast moving grains trap pores, but the average grain size in the ceramic affects important mechanical properties such as strength and fracture toughness [Figure 2-4]. Grain growth must be controlled in order to optimize mechanical performance. Additionally, abnormal grain growth, which can be severely detrimental mechanical properties, must be suppressed.

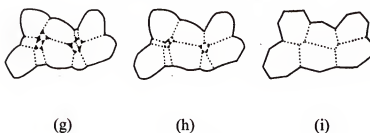
Initial Stage of Sintering



Intermediate Stage of Sintering



Final Stage of Sintering



KEY

..... Grain boundary

○ Grain

▼ Porosity

Figure 2-3. Changes That occur during Sintering: (a) Starting Particles, (b) Rearrangement, (c) Neck Formation, (d) Neck Growth and Volume Shrinkage, (e) Lengthening of Grain Boundaries, (f) Continued Neck Growth, and Grain Boundary Lengthening, Volume Shrinkage, and Grain Growth, (g) Grain Growth with Discontinuous Pore Phase, (h) Grain Growth and Porosity Reduction, (i) Grain Growth and Porosity Elimination [Rich92].

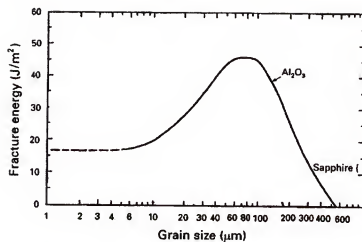


Figure 2-4. Fracture Toughness of Alumina vs. Grain Size at 22°C [Adapted from Rice96].

Grain growth occurs by the migration of grain boundaries. More loosely bound atoms of a crystal with a convex boundary seek to lower their potential energy by jumping to an adjacent crystal having a concave boundary [Figure 2-5]. This movement of atoms causes grain boundary migration in the opposite direction of the atomic jumps. In a microstructure composed of cylindrical grains of varying curvature, grains with greater than six sides grow by absorbing grains with less than six sides [Figure 2-6].

Grain growth can be controlled through proper selection of processing variables including firing time and temperature, or through the addition of coarsening prevention aids such as MgO.

Liquid-Phase Sintering

One of the powders (Coors AD85 alumina) used in this study to produce fired ceramic test specimens relies on liquid-phase sintering for densification. Liquid-phase sintering can be described as a sintering process where a portion of the material being sintered is in the liquid state [Bars97]. This sintering technique has two major advantages over solid state sintering. Liquid-phase sintering occurs much more rapidly than solid-state sintering, and it results in ceramic bodies with much more uniform densification. The reason for these advantages is that the liquid phase reduces the friction between particles, allowing them to move and rearrange more freely. Through exertion of capillary forces, the liquid also promotes rapid rearrangement of particles as well as the dissolution and reprecipitation of any sharp particle edges.

The liquid phase sintering process can be modeled as three stages [Figure 2-7]. The first stage is called particle rearrangement. Very rapid densification occurs during this stage due to the rearrangement of particles under the exertion of capillary forces, as

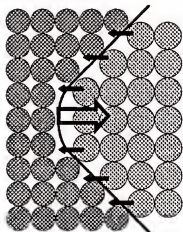


Figure 2-5. Atomic View of Curved Boundary. Atoms Will Jump from Right to Left, and the Grain Boundary Will Move in the Opposite Direction [Adapted from Bars97]

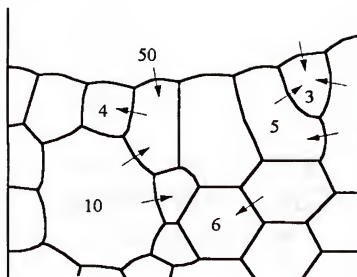


Figure 2-6. Grain Shape Equilibrium and Direction of Motion of Grain Boundaries in a Two Dimensional Sheet (the Grains are Cylinders in this Case) [Bars97].

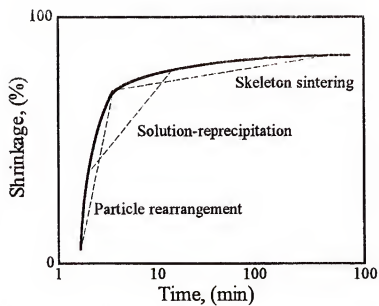


Figure 2-7. Time Dependence of Shrinkage Evolution as a Result of Liquid Phase Sintering Mechanisms [Bars97]

well as filling of pores by the liquid phase. The rate of shrinkage during this phase of sintering is modeled as [Mari87]

[Equ 2-1]

$$\frac{d\delta}{dt} = \frac{2\delta^3\gamma}{3\eta l^2 a}$$

where,

$d\delta/dt$ = the rate of grain center approach due to viscous flow (shrinkage rate)

γ = energy of the liquid surface

η = apparent viscosity of the liquid

δ = distance between grain centers

a, l = geometric parameters

It is evident from this equation that both the viscosity of the liquid phase and the surface energy of the liquid influence the densification rate of the ceramic body during the rearrangement process.

The fraction of complete densification provided by the rearrangement process is dependent on the volume fraction of liquid phase in the ceramic [King59]. The amount of shrinkage in the ceramic due to this mechanism is slightly greater than the volume fraction of liquid phase that is present. For a ceramic body with 0.25 volume fraction of liquid phase, the volume fraction of shrinkage by the rearrangement process is about 0.3. Assuming the ceramic had a green density of 60% theoretical, this first stage of sintering would account for densification up to 90% theoretical density. Above 90% relative density, the second and third stages would dominate densification.

The second stage of liquid phase sintering is solution-precipitation. Capillary forces increase the chemical potential of atoms at the points of contact between adjacent particles relative to the areas that are not in contact. A chemical potential gradient results

and induces the dissolution of the atoms at the contact points and their reprecipitation away from the area of contact. This process leads to continued shrinkage and densification.

Factors that increase the rate of densification during the process include increases in the surface energy of the liquid phase, and the diffusivity of the solid in the liquid [Mari87]. Specifically,

[Equ. 2-2]

$$\frac{dy}{dt} = \frac{3Dc\delta\gamma\Omega^2}{2kTl^2a}$$

where,

dy/dt = sintering rate

D = diffusivity of solid in the liquid

γ = surface energy of the liquid

Ω = atomic volume of solid phase atoms in the liquid

k = Boltzmann's constant

T = temperature

δ = distance between grain centers

a, l = geometric variables

c = constant

After a rigid skeleton is formed between the particles, the solid-state sintering process described earlier begins to dominate, and the densification rate is greatly reduced.

An Alternative to Conventional Firing

The energy required to drive both liquid and solid-state sintering of ceramics is normally supplied by conductive, convective and radiant heat from heating elements or through combustion of a fuel gas. An alternative technique to drive the sintering reaction is microwave heating. When conventionally sintered Coors AD998 alumina thick (20-50 mm) armor tiles were annealed at high temperatures using microwave energy to

enhance bonding between the grains and relieve residual stresses, there was a statistical increase in the hardness relative to that of the untreated tile [Kass94]. Though there was no statistical difference in the fracture toughness and flexural strength between the two cases, the standard deviations in these properties were significantly lower for the annealed tile than for the untreated tile. Ballistic tests performed on the tile showed that microwave annealing improved the ballistic performance for annealed tiles with thickness of 30 mm or greater. This improvement was attributed to a strengthening of grain boundaries through the removal of grain boundary precipitates.

If the same improvements can be generated with thin (<10 mm) alumina bodies using microwave heating for densification, then application of microwave processing could be extended to many other areas (cutting tools, thin armor tile, crucibles). A thorough literature review on microwave sintering is needed to provide insight into the technique and its potential as a candidate for processing thin alumina bodies.

Microwave/Material Interactions

Microwaves are electromagnetic waves with frequencies ranging from ~0.3 to 300 GHz [Clar96, Figure 2-8]. When a microwave strikes the surface of a material, three different scenarios are possible [Figure 2-9]. The microwave could pass through the material without attenuation, it could be reflected back from the surface, or it could penetrate into the volume of the material and experience attenuation [Sutt89]. Metals normally reflect incident microwaves. Ceramics, such as alumina, either transmit or partially absorb the incident microwaves depending on the frequency of microwave radiation, the temperature of the ceramic, and the ceramic in question.

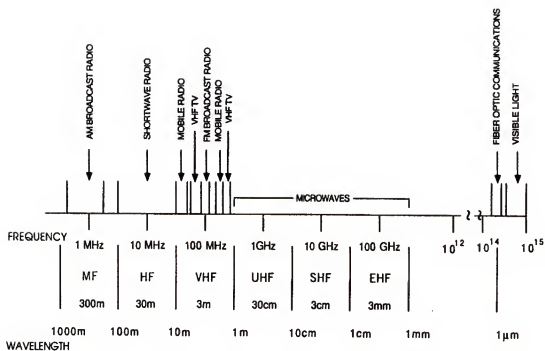


Figure 2-8. The Electromagnetic Spectrum [Scot93]

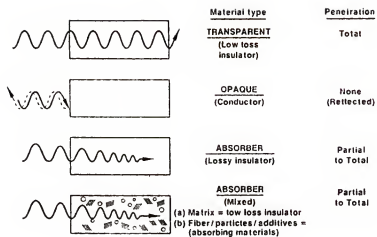


Figure 2-9. Interaction of Microwaves with Materials [Sutt89]

The type of macroscopic behavior of a ceramic in a microwave field is dependent on microwave/material interactions at the atomic and microstructural levels. Microwaves can interact with materials either through polarization or conduction processes [Clar96]. Polarization involves the formation and rotation of electric dipoles, while conduction requires the long range movement of electric charge. Inertial, elastic, and friction forces resist the induced motion causing losses and the electric field attenuation [Sutt89]. These losses cause the volumetric heating of the material.

At the lower microwave frequencies the losses are predominately due to ionic conduction [Figure 2-10]. However, as frequency increases other mechanisms begin to dominate. Since it is difficult to differentiate between the losses, they are usually grouped together and termed effective losses, ϵ_{eff}'' .

Equations

Several equations have been developed to model a material's response to an applied microwave field. These equations provide useful insights between the physical parameters such as microwave frequency, level of power applied, a material's dielectric properties, and amount of microwaves absorbed by a material.

The complex permittivity, ϵ^* is often used to describe the response of a material to microwave radiation. The complex permittivity of a material is defined as [Sutt89]

$$\epsilon^* = \epsilon_0 (\epsilon_r' - j\epsilon_{\text{eff}}'') \quad (\text{Equ. 2-3})$$

where,

ϵ_0 =the permittivity of free space (8.86×10^{12} F/m)

ϵ_r' = the relative dielectric constant

ϵ_{eff}'' =the effective relative dielectric loss factor

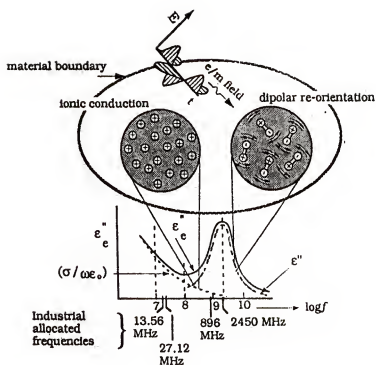


Figure 2-10. Effective Loss Factor Due to Dipolar Ionic Conduction [Meta93]

The loss tangent, $\tan \delta$, is another important measure of a material's response to incident radiation. The loss tangent is a measure of the energy dissipated within a material relative to the energy stored within a material. It is defined as [Sutt89]

$$\tan \delta = \frac{\frac{\epsilon''}{\epsilon'}}{2\pi f \epsilon'} = \frac{\sigma}{2\pi f \epsilon'} \quad (\text{Equ.2-4})$$

where,

σ =the total effective conductivity (S/m) caused by conduction and displacement current

f =the frequency of microwave radiation

The relative magnitudes of ϵ_r' and $\tan \delta$ are dependent on the frequency of the incident microwave radiation, as well as the temperature of the material. Typical values of ϵ_r' and $\tan \delta$ for a commercially available polycrystalline alumina at room temperature and at a frequencies from 1-3 GHz are 9.95, and 0.01, respectfully [Batt95]. At 1400°C, the value of ϵ_r' for the alumina increased to near 12, while $\tan \delta$ increased nearly an order of magnitude to 0.08.

The power absorbed by a body subject to microwave radiation is [Sutt89]

$$P = \sigma |E|^2 = 2\pi f \epsilon' \tan \delta |E|^2 \quad (\text{Equ. 2-5})$$

where,

E =the root mean square of the electric field within the material in [V/m]

P =the power absorbed per unit volume of material [W/m^3]

The power is absorbed both at the surface of the material and inside the volume of the material. The depth of penetration of microwaves into the material can be modeled

by [Clar96]

$$D = \frac{\lambda_0}{2\pi(2\epsilon_r')^{1/2}} \left[\left(1 + (\tan \delta)^2 \right)^{1/2} - 1 \right]^{-1/2}$$

(Equ. 2-6)

where,

D =penetration depth at which the incident power is reduced by 1/e
 λ_0 =the incident or free space wavelength

The depth of penetration into a sample and the overall power absorbed by the sample are very important considerations when microwave sintering samples of alumina, as well as other materials.

Microwave Sintering of Alumina

Since the late 1980s, there have been a number of studies on microwave sintering of alumina. These studies have compared the densification, mechanical properties, and microstructures of conventionally and microwave sintered specimens of a number of different aluminum oxide powders. They have employed techniques such as microwave hybrid heating and stand-alone microwave sintering, and used both single-mode and multimode cavities for processing. Sample size has varied from as small as two centimeter diameter thin disks to as large as meter long rods, while batch sizes have been as few one sample per run to as many as 108 per run. An overview of the studies is presented in Table 2-1.

Almost all studies listed in the table have been performed on high purity alumina (99%+ purity). The vast majority of studies in the literature appeared to have focused on high purity alumina, with only a few studies done on alumina with 92-98% purity

Table 2-1. Summary Information on Some Microwave Sintering Studies on Alumina

Study	Alumina	Microwave	Hybrid?	Samples/Batch
Tian88	Baikowski CR15, CR20, CR30	2.45 GHz single mode applicator	No	1
De90	Alcoa A-16, Sumitomo AKP-15, AKP-30, AKP-50	2.45 GHz multimode cavity	Yes	1
Katz91	AKP-50	2.45 GHz single mode cavity	Yes	20
Pati91	Alcoa A16SG, Sumitomo AKPO-30MG	2.45 GHz single mode	No	1
Jann91	Sumitomo AKP-50	28 GHz multimode cavity	No	1
Patt91	Alcoa A-16, Baikowski CR30	2.45 GHz multimode cavity	Unclear	1-40
Chen92	99.8% pure MgO doped	2.45 GHz single mode cavity	No	1
Bran92	Alcoa A100SG	2.45 GHz multimode cavity	Yes	1
Samu92	Alcoa A100SG	2.45 GHz multimode cavity	Yes	3
Brat94	debased alumina	2.45 GHz multimode cavity	Yes	108
Chen95	Fisher High Purity Alumina	2.45 GHz multimode cavity	No	1
Vode96	Alcoa A16SG, and CT800SG	2.45 GHz multimode cavity	Yes	1
Lee97	Sumitomo AKP-30	2.45 GHz single mode cavity	Yes	6
Flif98	Sumitomo AKP-50	35 GHz single mode	No	1
Xie98	Ceralox 99.97% pure APA	2.45 GHz multimode cavity	Yes	1

[Sutt88, Bert76]. There does not appear to have been any studies on microwave firing of low-purity alumina (less than 90% pure) where liquid phase sintering plays a major role in sintering. Because of the lack of information on microwave sintering of low-purity alumina, as much information as possible was gleaned from the results of the high-purity alumina studies.

Features of Microwave Sintering

Features inherent to microwave sintering differentiate it from conventional sintering and lead to potential enhancements of the sintering process. These features include volumetric heating, increased diffusion rates, and the capability of microwaves to preferentially target porous regions of ceramic materials [Bran92, Jann91, Penn97].

The capability of microwaves to dissipate energy into the volume of a material is dependent on the depth of penetration of the incident radiation. Using equation 2-6 and the data from [Batt95], the depth of penetration for polycrystalline alumina at room temperature is approximately 62 cm at room temperature and decreases to 7 cm at 1400°C.

The volumetric heating phenomenon has important ramifications on the temperature profile existing within a material during heat up or cool down. Examples of the temperature profiles that can be produced by conventionally and microwave (hybrid) heating of identical alumina samples are shown in Figure 2-11. The conventional sample shows the typical temperature profile of a low thermal conductivity ceramic. The temperature near the outside surface is considerable hotter than the sample interior. The microwave hybrid heated sample, however, shows a variable relationship between position within the sample and temperature.

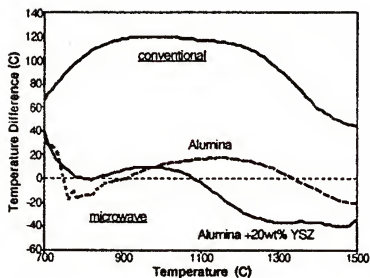


Figure 2-11. Comparison Volumetric Data for Alumina Heated at 10°C/min to 1500°C [Bran92]. The Temperature Difference Expressed in the Figure is the Difference in Temperature between the Surface and Center of a 5 cm Diameter by 6 cm Thick Cylinder of the Given Materials

In addition to this volumetric heating, faster densification has also been partially attributed to enhanced diffusion during microwave sintering. An example of the latter has been provided for alumina at 28 GHz [Figure 2-12]. As the figure suggests, the apparent activation energy for microwave sintering was greater than 3.5 times that for conventional sintering for the conditions in the study [Jann91].

Several other studies have cited enhanced diffusion in a microwave field as a cause for experimentally observed increases in densification or other diffusion dependent processes [Jann88, Chen92, Samu92, Will94, Wroe96, Nigh96, Boch97, Wils97, Xie98]. All of these experiments have been performed on materials that do not rely on liquid-phase sintering for densification. To achieve the enhancements observed in the studies, microwaves must increase the rate of densification-related diffusion (grain boundary, or volumetric diffusion) relative to that of coarsening-related diffusion (surface diffusion).

A study by Samuels et. al. [Samu92] on microwave sintering of alumina and alumina-zirconia composites suggested that a clear reduction in activation energy for grain boundary self-diffusion occurred during microwave processing. However, Nightingale et. al. [Nigh96] inferred that microwaves enhance lattice diffusion more than surface or grain boundary diffusion.

Though it was not clear which densification-related diffusion mechanism was actually enhanced by microwaves, there has been growing evidence that the microwave field induces an additional driving force for diffusion in solid-state sintering of ceramic materials [Ryba94]. According to Wroe and Rowley [Wroe96] this driving force could be due to the accumulation of space charge at grain boundaries. The alternating electric field could establish an electric gradient within the grains of a low electric conductivity

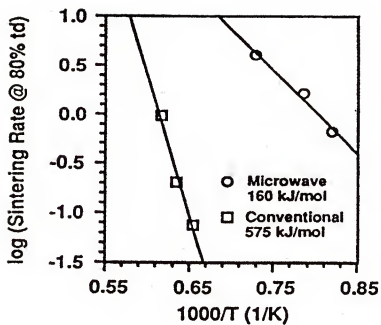


Figure 2-12. The Apparent Activation Energy of Sintering High Purity Alumina for Microwave Firing and Conventional Firing at 28 GHz [Jann91].

ceramic (alumina, zirconia) by inducing a net polarization around dipoles [Figure 2-13]. This could reduce the activation energy required for diffusion and lead to the enhancement of differential sintering [Will94].

More recent experimental studies by Rybakov et al. [Ryba97] and Janney et al. [Jann97] have provided some direct evidence for the non-thermal influence of microwaves on mass transport in solids. Rybakov et. al. [Ryba97] have found that microwaves induce currents inside single crystals of silver chloride and sodium chloride. -The time scale for the relaxation of the response was consistent with the ionic diffusion, and the polarization phenomenon. Janney et. al. [Jann97] have found enhanced diffusion in single crystal alumina during microwave heating.

When the electric field inherent to microwaves interacts with free surfaces or interfaces between phases, an additional driving force for diffusion is created [Boos97]. This driving force has been named the "ponderomotive" driving force [Ryba94]. For strong microwave field strengths, the ponderomotive force can compete with thermochemical driving forces that are present during sintering of ceramics [Boos97].

In local areas within the ceramic, such as on the surfaces of pores with convex surfaces [Will97], the electric field strength can be extremely high relative to the spatially averaged electric field strength [Calm97]. Since diffusion flow rates are proportional to the square of the absolute value of the electric field strength, the flow rates can be very high locally [Calm97]. The local field strength may even be high enough to ignite plasma which could provide mass transport by evaporation or enhance surface diffusion [Calm97, Will97].

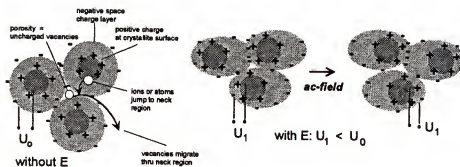


Figure 2-13. Periodic Reduction of the Potential Barrier for Vacancy Flow from the Pore Region through the Neck Region by polarization of the Space Charge Layer by an Alternating Microwave Field [Will96].

Evidence for the targeting of porosity by microwaves has been provided by the large increase in the loss tangent with increasing porosity [Figure 2-14]. For a constant electric field, the increase in this ratio translates into increased power absorbed by the sample (as shown in Equ. 2-5) during the initial stages of sintering.

Densification

On the surface, microwave sintering of alumina appears very similar to conventional sintering. The relative rates of densification have similar dependence on particle size and distribution of the starting powder [De90]. The sintering curves generally have similar shape, and sintered samples are generally similar in appearance. However, there is at least one notable trend that suggests that microwave densification is somewhat different than conventional sintering. Sintering of alumina by microwaves enhances densification compared to conventional sintering under identical conditions. This enhancement manifests itself as a reduction in the required soak temperature to reach a given relative density. The degree of enhancement ranges from 50-300°C and depends on the microwave sintering frequency, the sintering temperature, and powder being processed. An example of a sintering curve that shows this enhancement is given in Figure 2-15.

The dominant factor influencing the reduction in required soak temperature is the microwave sintering frequency. The sintering frequency influences the rate of power absorbed directly and indirectly through the loss tangent [Equ. 2-5]. Figure 2-16 plots the loss tangent of Coors AD995 alumina as a function of frequency. As the microwave frequency changes from 2.45 GHz to 28 GHz the loss tangent increases about 250%.

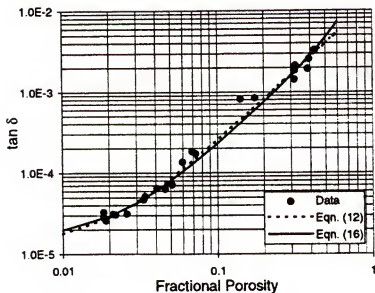


Figure 2-14. Loss Tangent Vs. Fractional Porosity for an Alumina Body at 9 GHz [Penn97]. Note: The Equation Numbers in the Box Do Not Correspond to Equations in the Dissertation.

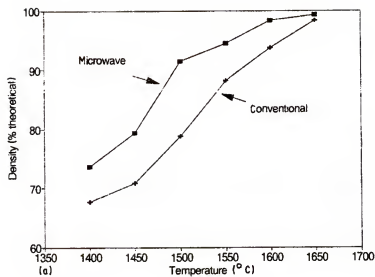


Figure 2-15. Density as a Function of Temperature for Alcoa A1000SG Alumina Powder for Sintering in a 2.45 GHz Microwave Oven [Samu92].

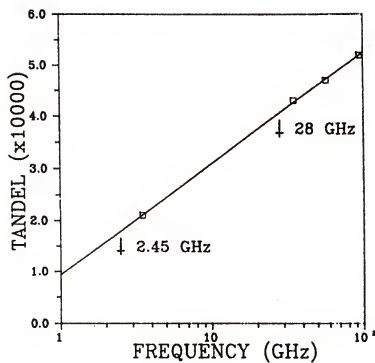


Figure 2-16. Variation of Loss Tangent with Frequency at 25°C for Coors AD995 Alumina [Jann92].

Combining this increase with the direct influence of frequency on power absorbed yields a total increase in power absorbed at 28 GHz of 25 times that of 2.45 GHz.

For a given microwave sintering frequency, there is still some variation in the magnitude of microwave enhancement over conventional sintering. The variation is due to dependence of the loss tangent on the sintering temperature and the powder composition. Figure 2-17 is an example of how the loss tangent depends on temperature.

The composition of sintered powder also influences the relative magnitude of microwave enhancement over traditional sintering. Compacts made from highly agglomerated powder seem to provide less relative enhancement than compacts made from powder with low level agglomeration [Vode96]. The relative decrease in required processing temperature for compacts of 2.45 GHz microwave sintered alumina with low and high agglomeration is 100°C and 60°C, compared to conventionally processed samples [Vode96].

The combination of sample volumetric heating, enhanced diffusion inherent to microwave sintering, and ability of microwaves to target porosity also has some unique and desirable effects on the alumina sintering and microstructure development.

Microstructure

Microwave sintering can result in different microstructural development and final microstructure in alumina bodies relative to that found using conventional sintering. A number of different studies have examined various aspects of microstructure produced in alumina bodies after microwave sintering. These have included grain growth rate, grain size and distribution, and grain morphology.

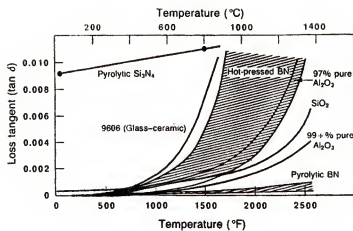


Figure 2-17. The Loss Tangent (8 to 10 GHz) Versus Temperature [Sutt89]

Grain growth of alumina during microwave sintering appears to be controlled by a lattice diffusion mechanism. A study by Cheng [Chen91] on grain growth of experimentally sintered alumina in a single-mode TEM₁₀₃, 2.45 GHz microwave found that grain growth rate could be modeled as

$$\frac{dG}{Dt} = \frac{CD}{G(1 - \rho)^2} \quad (\text{Equ. 2-7})$$

where,

G=Grain Size

D=Diffusion Coefficient

T=Time

C=constant

ρ =bulk density

This model is very similar to that used to model grain growth in conventional sintering at temperatures where lattice-diffusion-controlled mechanism controls grain growth. The study also found that an acceleration of grain growth in microwave sintered alumina is more likely to occur with longer hold times than higher temperatures.

One possible reason for the dominance of the lattice diffusion mechanism is provided by Tian and Johnson [Tian88]. They speculated that small grain size in microwave sintered alumina was due to a rapid transition to higher temperature that avoids surface diffusion in the lower temperature regime and is dominated by grain boundary and lattice diffusion.

Grain size/relative density relationship

Despite the possible dominance of lattice diffusion in microwave sintering, it is unclear whether or not microwave sintering produces alumina bodies with smaller grain size for a given relative density compared to conventionally processed samples. A few studies found that microwave sintering does indeed result in alumina bodies with smaller grain size for a given relative density [De90, Patt91, Tian88]. These studies were done using 2.45 GHz microwave radiation in single mode and multimode cavities. Later studies by Xie using 2.45 GHz radiation, and Fliflet using 35 GHz show that the grain size/relative density relationship is the same for conventional sintering[Xie98, Flif98]. Representative results are provided in Figures 2-18 and 2-19.

One possible explanation for the different trends is provided by De's work [De90]. De found that the relationship between grain size and relative density is dependent on the rate the alumina samples were heated. The samples with both the highest relative density and smallest grain size are those heated to the soak temperature at the fastest rate of 750°C/minute [Figure 2-20].

Microstructural spatial uniformity

It is possible to produce microwave sintered alumina bodies with better microstructural uniformity than with conventional sintering. Several studies using 2.45 GHz microwave radiation for sintering produced alumina bodies with good spatial uniformity in grain size [De90, Katz91, Patt91, Brat94, Bran95]. Examples of this uniformity are shown in Figures 2-21 and 2-22. This uniformity is due to good temperature and electric field uniformity during sintering.

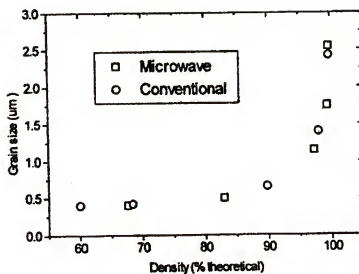


Figure 2-18. Grain Sizes Vs. Density for Two Sintering Methods [Xie98]

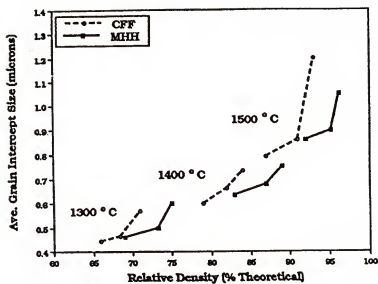


Figure 2-19. Grain Size Vs. Relative Density for Microwave Hybrid Heating and Conventional Fast Firing [De90].

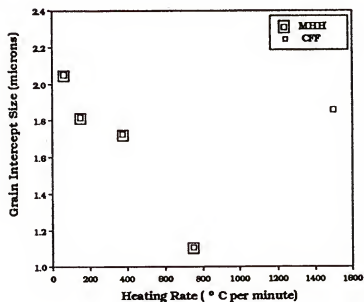
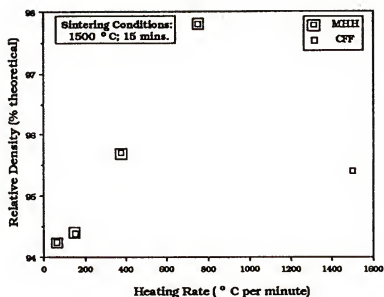


Figure 2-20. Effect of Heating Rates on the Sintering of Sumitomo AKP-50 Alumina [De90]. Note: MHH=Microwave Hybrid Heating, CFF=Conventional Fast Firing.

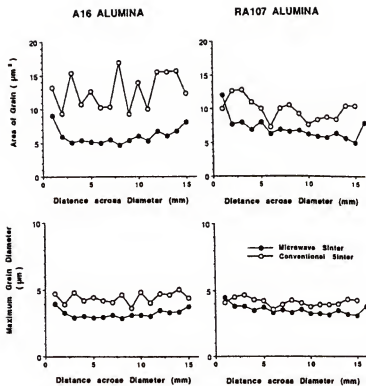


Figure 2-21. Average Grain Area and Maximum Grain Size as a Function of Position across the Pellet, for Microwave and Conventionally Sintered A16 and RA107 Alumina [Patt91].

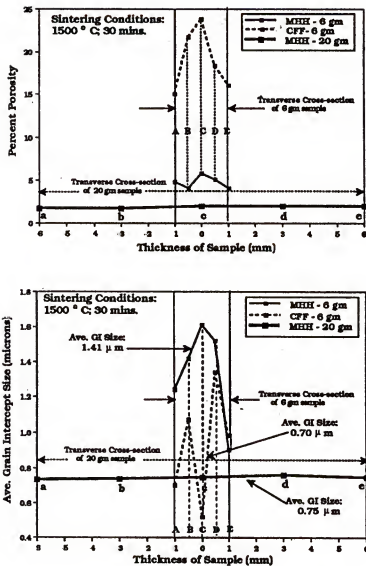


Figure 2-22. Microstructural Uniformity Comparisons for Microwave (Hybrid) Heated and Conventionally Fast Fired Alcoa A-16 Samples Sintered at 1500°C for 30 minutes [De90]. Note: MHH=Microwave Hybrid Heating, CFF=Conventional Fast Firing.

When a large gradient in temperature or electric field exists within an alumina body during sintering, the microstructural homogeneity disappears. A few studies [Tian88, Patt91, Pati91] found that there were larger grains, perhaps even extremely larger grains, in the center of the microwave sintered body than at the surface of the sample. They attributed this to temperature gradients, possibly due to short sintering times, or high electric field intensity at the center of the samples.

In order to produce alumina bodies with microstructural uniformity, control of both the temperature and electric field profiles must be achieved. A further consideration is the thickness of the sample to be sintered. As Figure 2-22 illustrates, samples with different masses that are microwave sintered under similar conditions can produce bodies with varying microstructural uniformity.

Grain Morphology

In addition to improved microstructural uniformity, two studies found that the grains of microwave sintered alumina bodies are more equiaxed than those of conventionally sintered bodies [Patt91, Vode96]. This is a result of microwave sintered bodies having a much higher open porosity during the intermediate stage of sintering, which afford more regular grain growth.

This difference in grain morphology could have implications to the fracture toughness of the resulting alumina specimens. More equiaxed grains should result in less microcracking due to a reduction in the thermal mismatch stress. This could reduce fracture toughness in the body.

Mechanical Properties of Microwave Sintered Alumina

Literature suggests that it is possible to produce microwave sintered samples with at least as good of mechanical properties as conventionally fired samples, and possibly better. Table 2-2 summaries a few mechanical property studies on microwave sintered alumina. On average, the mechanical properties of microwave processed alumina seemed to be fairly equivalent to those of conventionally processed alumina when compared at similar densities.

There are two cases where the microwave processed alumina samples had either increased fracture toughness or hardness [Kass94, Patt91]. The case of increased hardness was attributed to grain boundary strengthening possibly through the volatilization of impurities at the grain boundaries. In the case where there was improved fracture toughness, it was speculated that microwave sintering produces more thermal mismatch between grains and grain boundaries, causing extensive microcracking. This is despite the microwave samples having more equiaxed grains than the conventionally produced samples.

There was one case (not in table) where the strength was lower for the microwave processed samples [Apte92]. The processed samples for this case were large, thick slabs. The decrease in strength was attributed to significantly weaker grain boundaries in the microwave fired alumina. Large, discontinuous grains were observed on fracture surfaces of the microwave processed samples. These discontinuous grains were perhaps a result of long sintering times in combination with a non-uniform temperature distribution throughout the cross section of the sample.

Table 2-2. Summary of Studies on the Mechanical Properties of Microwave Processed Alumina Bodies

Study	Alumina	Microwave	Sample Size	Avg. Density	Result
Patt91	99.8% pure Baco RA107 and Alcoa A16	Multi-mode 2.45 GHz	Pellet- 19 mm Dia. 16 mm Ht.	99+%	MW samples had comparable strength and hardness to conventional, but higher toughness
Lee97	99.99% pure Sumitomo AKP30	Single Mode 2.45 Ghz w/ Zirconia Casket	pellet- 2.2 cm Dia., 0.2 cm thick	99.5 %	Hardness consistent w/ conventional values, fracture toughness lower
Iska91	?	Multi-mode 2.45 GHz	pellet- 2.3 cm Dia., 0.7-0.9 cm thick	98.25%	Fracture toughness ~ 4.5 MPam ^{1/2}
Kass94	Coors AD995	28 GHz	Squares- 15.2 cm square x 20-50 mm thick	99%	Hardness higher in MW treated samples, other properties equivalent
Apte92	99.8% pure Baco RA107 and Alcoa A16	Multi-mode 2.45 GHz	Slab- 37 mm square x 90 mm ht.	99%	Strength less in MW processed samples

Spatial uniformity of mechanical properties across the body appears to be good in the cases of microwave processing. Fracture toughness and hardness were determined to be very uniform across the cross-section [Figure 2-23] and along the diameter of the microwave fired alumina specimens [Lee97].

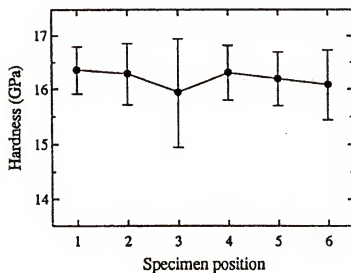
Microwave sintering can produce sintered bodies that have spatially uniform and equivalently strong mechanical properties as those produced via conventional firing. However, care must be taken to avoid discontinuous grain growth by limiting soak times and properly controlling the temperature profile within the body.

Microwave (Hybrid) Heating

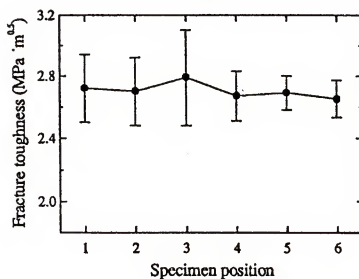
It is necessary to use microwave hybrid heating to fire alumina at low microwave frequencies in multi-mode cavities when processing from room temperature. These hybrid systems surround the alumina sample with a material that readily absorbs the incident microwave temperature at room temperature and beyond. This absorbing material is commonly referred to as a susceptor.

There are two basic categories of susceptors found throughout the literature. The categories include consumable and permanent susceptors. Consumable susceptors include carbon felt and binders, which burn off at higher temperatures [Vode96]. This type of susceptor only serves to preheat the sample to temperature where it can absorb the incident microwaves. The sample is then exposed to the full wattage of the incident radiation. This can create an inverted temperature profile within the sample, potentially leading to wide spatial variations in grain size.

Permanent susceptors do not burn off at higher temperatures, but remain intact through the entire firing processes. Common permanent susceptors used throughout the



(a)



(b)

Figure 2-23. (a) Hardness and (b) Fracture Toughness for Batch Processed Alumina Specimens in Terms of Specimen Position. Error Bars Represent Standard Deviations [Lee97].

literature include silicon carbide and silicon carbide based composites [Cozz96, Xie98, De90], zirconia fiber boards [Katz91, Samu92], and others [Bran92, Brat94]. Permanent susceptors absorb some of the incident microwave radiation and convert it into heat used for warming the sample. At the upper temperatures where the alumina sample efficiently couples with the remaining radiation, there are two potential sources for heating the sample. These include microwave volumetric heating and infrared and conductive heating from the susceptor [Figure 2-24]. Through proper tailoring of these two sources of heating, a uniform temperature distribution can be achieved throughout the sample.

A potential method to tailor the ratio of microwave to infrared/conductive heating reaching the sample is by varying the amount of absorbing phase in composite susceptors. Cozzi studied the microwave dielectric properties of high alumina cement/silicon carbide particle susceptors over a range of temperatures [Cozz96]. The study found that the dielectric properties of these susceptors at high temperatures varied significantly with silicon carbide content. A summary of those findings is presented in Table 2-3, and Figures 2-25 and 2-26. Since the loss tangent and the relative dielectric constant both affect the amount of power absorbed by the susceptor, there should exist an optimal silicon carbide content where balance is achieved between the two sources of heating. This should lead to uniform temperature in the sample during firing and the production of samples with uniform microstructure [Figure 2-27].

Because of the proven track record of these alumina cement/silicon carbide particle susceptors, and ease of altering dielectric properties through control of the absorbing phase, these materials were selected for the fabrication of susceptors for this

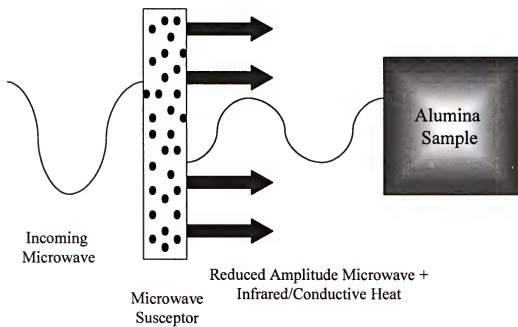


Figure 2-24. Microwave Hybrid Heating

Table 2-3. Dielectric Properties of Susceptors at 1200°C [Adapted from Cozz96]

% SiC	ϵ_r'	$\tan\delta$
10	5.4	0.045
20	7.2	0.05
30	8.5	~0.12

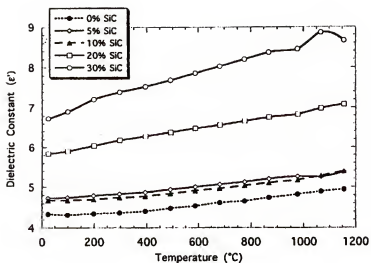


Figure 2-25. Dielectric Constant, Measured at 2.46 GHz, Vs. Temperature for Several Compositions of Susceptors [Cozz96].

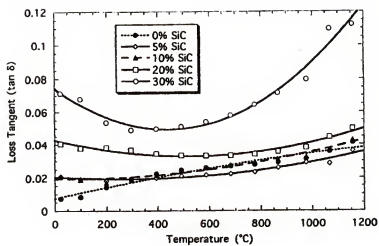


Figure 2-26. Loss Tangent for Several Susceptor Compositions at 2.46 GHz [Cozz96].

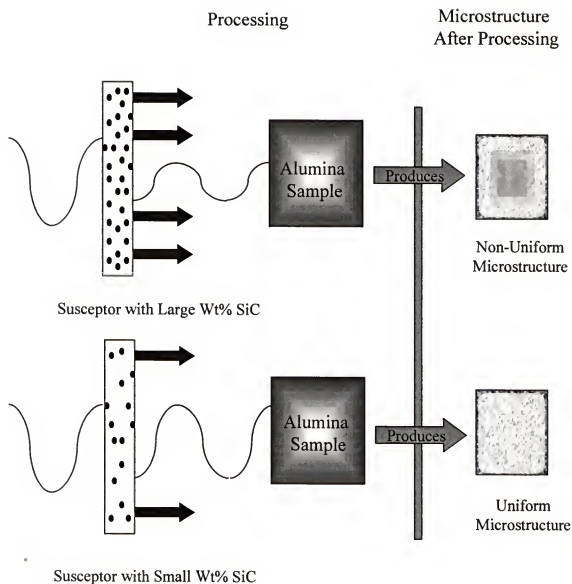


Figure 2-27. Possible Effect of Silicon Carbide Weight Percent in Susceptors on Processing and Final Microstructure of Alumina Body.

study. The development of the susceptors into useful components for microwave hybrid heating is the focus of the next chapter.

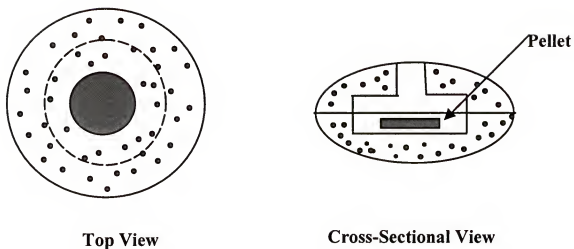
CHAPTER 3

SUSCEPTOR AND INSULATION DESIGN

The processes for selection and design of system insulation and susceptors were ones of gradual evolution. A total of not less than seven stages were completed before arriving upon the final susceptor design, while at least three stages were completed in the evolution of system insulation. Each new design iteration either added one or more new features considered important to the overall design, or provided possible solutions to inadequacies in previous designs.

Considerations in the development of the susceptors focused not only on the physical assets of the susceptor, but also on the alumina tiles that were sintered in them. Susceptor physical parameters considered important included size, compactness, and reusability. The susceptor design was further constrained by the desire to sinter a large batch of 2" x 2" x 3/8" thick alumina tile at 1600°C for 1 hour using 3200 W of 2.45 GHz microwave radiation. Additionally, all tiles had to be sintered to a similar percent theoretical density and possess similar final microstructures.

The path chosen to reach the final design goals was one of gradual scale-up in both the size of the sintered alumina body and the number of tiles sintered per batch. The first arrangement was only capable of sintering small 1 in² top surface area pellets [Figure 3-1]. It consisted of a composite made from a base of Alfrac 66 cement with the



- *Good For only 1 in² pellets
- *Used for Start-up
- *Both top and bottom halves doped with coarse silicon carbide (10-50 wt%)

Figure 3-1. Phase One Susceptor Design

remaining 10-50 wt% being composed of microwave absorbing 1-2 mm diameter α -silicon carbide particles. Its basic function was to provide a medium for evaluation of the microwave sinterability of various commercially used alumina powders. Relative density vs. soak time curves for pellets of three different alumina powders sintered at 1450°C in a 30 wt% SiC susceptor using 1600 W of 2.45 GHz microwave radiation are shown in Figure 3-2. Notice that the Coors AD998 alumina had the least sinterability of any of the powders tested.

Further phases in susceptor system development dealt with issues such as tile scale-up, and single tile to batch processing. Problems such as tile stickage and warpage were also overcome. The phase four susceptor is an example of a system that could sinter multiple tiles at once [Figure 3-3]. One tile was placed in each square susceptor enclosure and supported by a large thin square of Alfrax 66 cement. These compartments were stacked on top of one another to accommodate three tiles per batch. Temperature was monitored via an optical pyrometer that observed the top surface of the top tile through a circular hole in the Alfrax 66 "roof".

The bottom and middle tiles were fired to similar relative densities during processing. The top tile was considerably less dense after sintering than the other two tiles. This was primarily due to heat losses out of the pyrometer view hole.

The final susceptor design was that of a long tube with square cross-section [Figure 3-4]. The single piece design had advantages over previous designs in areas such as simplicity and ease of fabrication. It also contributed to the uniform distribution of radiative and microwave energy to all tiles in the stack. The major trade-off was the loss

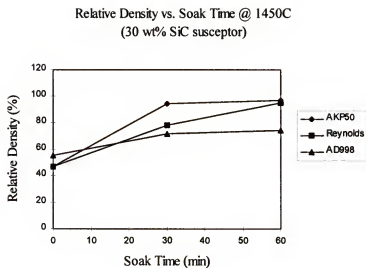


Figure 3-2. Densification Curves for some Commercially Available Aluminas Densified through Microwave (Hybrid) Heating.

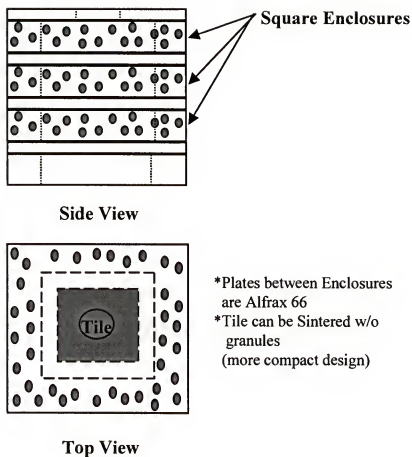


Figure 3-3. Phase Four Susceptor Design

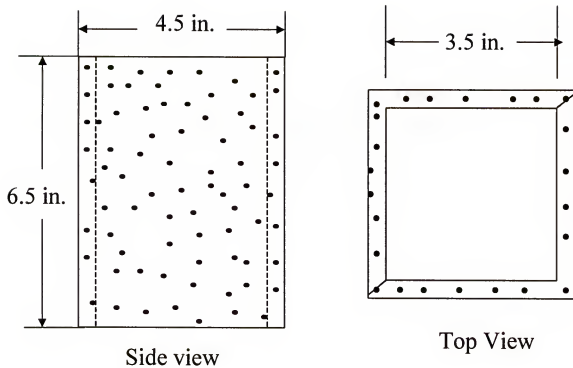


Figure 3-4. Final Susceptor Design

of system processing variables. For example, with the phase four design it was possible to place susceptors of varying weight silicon carbide at different levels in the stack. This was not possible with the final design. However, it was decided that the simplicity afforded in the final design was necessary in such a fundamental study.

Concurrent to the development of susceptor systems for sintering tile was the selection of insulation to contain the heat that the susceptors and tile bodies generated. Major considerations in selection of insulation for the susceptor system were system structural, thermal, and dielectric properties as well as cost. A good insulation system had to be compact, reusable, simple, rigid, and standardized. It must also have a low thermal conductivity and the capability to withstand the hostile microwave operating environment of 1500°C+ for periods in excess of 1.5 hours. In addition to the thermal and structural requirements, operation within a microwave environment dictated the need for low microwave absorption throughout the temperature range. An insulation that absorbs microwaves would act to screen the susceptors and samples from microwave radiation, thereby lowering efficiency. It would also transfer a substantial amount of heat into the surrounding environment. To avoid these problems, the material must have a low loss tangent.

Table 3-1 summarizes important thermal and mechanical properties of insulation selected to fulfill the needs of the project. Two of the insulation types selected were rigid and could be used in a structural capacity. They are listed with their modulus of rupture (MOR). The other insulation types are flexible and non load bearing.

Of the two structural insulations, the 174/400 fiberboard was judged best for high temperature microwave operations. It or an equivalent has been used satisfactorily in a

Table 3-1. Important Thermal and Structural Properties of Selected Insulations

Insulation	Producer	Max. Service Temp (°C)	Thermal Conductivity (W/mK)	MOR (MPa)	Composition
K3000 Firebrick	Thermal Ceramics, Inc.	1650	0.58 @ 1315°C	1.8 (18.2 kg/cm ²)	61% Al ₂ O ₃ 36.5% SiO ₂ ~2.5% other
KVS 174/400 Fiber Board	Rath Performance Fibers, Inc.	1650	0.33 @ 1500°C	8	81% Al ₂ O ₃ 19% SiO ₂
Alumina Mat	Zircar, Inc.	1650	0.30 @ 1425 °C	N/A	95% Al ₂ O ₃ 5% SiO ₂
Alumina Paper APA-1	Zircar, Inc.	1650	0.29 @ 1500°C	N/A	95% Al ₂ O ₃ 5% SiO ₂

number of other works on microwave sintering (proven track record). It had a lower thermal conductivity near the sintering temperature and was more reusable due to a higher resistance to thermal shock. It was also purer in composition with fewer trace elements that could heat when exposed to 2.45 GHz microwave radiation (i.e. Fe_2O_3). Its main disadvantage was cost. An 18" x 24" x 1" board priced around \$320, while a K3000 firebrick was ~\$6 per 9.5" x 4.5" x 2.5" brick.

The non-structural insulations were fairly equivalent in thermal properties and purity. However, the alumina mat was more cost effective. An 18" x 24" x 1" piece of alumina mat was \$80, while a 1/16" piece of APA-1 alumina paper of similar size was ~\$26.

A typical insulation system employed a structural insulation plus one or more kinds of non-structural insulation. The first insulation system used combined K3000 firebrick and alumina mat [Figure 3-5]

For this system, the "hamburger-shaped" microwave susceptor was placed in a cavity drilled at the joints of four K3000 firebricks stacked two bricks per level. Additional susceptor insulation was provided by alumina mat which lined the walls of the cavity. A top hole through the susceptor and insulation assemblies was used as a viewport for temperature measurement of the alumina tiles by a two color optical pyrometer.

The insulation was good for initial trials on microwave sintering of tiles and pellets. It was relatively inexpensive, and could be fabricated in a relatively short time. However, it had a number of deficiencies that prevented it from being used as the final insulation system. One consistent problem was the need to replace the alumina mat after

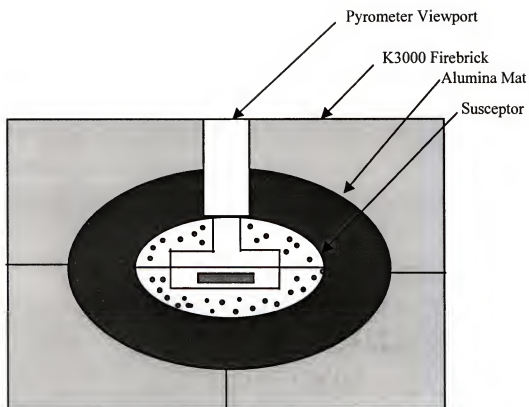


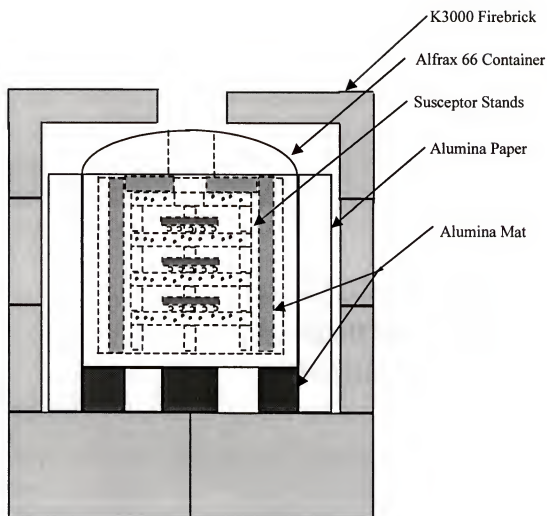
Figure 3-5. Cross-Sectional View of First Insulation System

each sintering run due to densification caused by direct contact with the extremely hot susceptor assembly. This need for replacement reduced the possibility of standardization between sintering runs. Densification of the alumina mat also created other problems such as a change in the thermal properties of insulation during sintering, and damage to the firebrick directly under the susceptor. In addition to densification related problems, the fire bricks had a tendency to thermally shock and the system could only accommodate one tile per run.

Further iterations in insulation selection attempted to find practical solutions to the need for standardization and repeatability between sintering runs, and scale-up to multiple tiles per experiment. Figure 3-6 shows the first attempt to provide a solution to those needs.

Susceptor stands were contained within an Alfrax 66 alumina based cement container. The container had an inner lining of alumina mat to minimize heat transfer out of the chamber. The outer wall of the chamber was surrounded by alumina mat and APA-1 paper as shown. Additional Alfrax 66 support legs were added to the assembly to support the chamber above the surface of the bottom fire bricks. The whole assembly was then encased by an outer shell of K3000 fire brick. Temperature of the surface of the top tile was again monitored with a 2 color IR pyrometer that observed the tile through a hole in the top of the assembly.

The system had a number of advantages over its predecessor. The addition of the Alfrax 66 support legs minimized direct contact of the chamber with the bottom firebricks, thereby reducing the area of brick damage. The design also was able to sinter 3-4 tiles per run, and was relatively inexpensive as far as material cost.



SIDE CUT-AWAY VIEW

Figure 3-6. Insulation Assembly for Sintering Multiple Tiles

Despite these improvements, there were a number of shortcomings that prevented the arrangement from being selected for the final insulation assembly. Thermal shock in the firebrick and Alfrax 66 chamber was prevalent, as was densification of the alumina mat in the inner chamber. The design only accommodated 3-4 tiles per run, with the top tile considered scrap. It was also very complex and tedious to assemble.

The final assembly selected for insulating and sintering the alumina tiles is shown in Figure 3-7. It combines the susceptor tube [Figure 3-4] and insulation into one compact rectangular sintering chamber. The walls of the insulation chamber consisted of 1" thick Rath KVS 174/400 fiberboard panels with linings of alumina mat to minimize heat transfer from joints between panels.

Sintering was performed on green tiles that were stacked on 3" x 3" x 1" tall Rath KVS 174/400 fiber board setter plates and then enclosed by the susceptor and insulation assembly.

An Omega "B" type thermocouple with an ungrounded junction, designation XPA-P30R-U-062-30-M-SX-6, was used to monitor temperature in the experiments. It was encased in a platinum-rhodium sheath and surrounded by MgO insulation. The sheath diameter was 0.062" and the maximum use temperature was 1650°C.

The thermocouple sheath was well grounded to the bottom of the microwave cavity to prevent any interactions with the microwave. The grounding was accomplished using the assembly shown in Figure 3-8. The assembly consisted of a 1/4" diameter bolt with a 7/64" diameter hole drilled through the center of the length of its shaft. The thermocouple was inserted into the shaft and soldered into place. The assembly was then inserted through a hole drilled in the bottom of the microwave processing cavity and

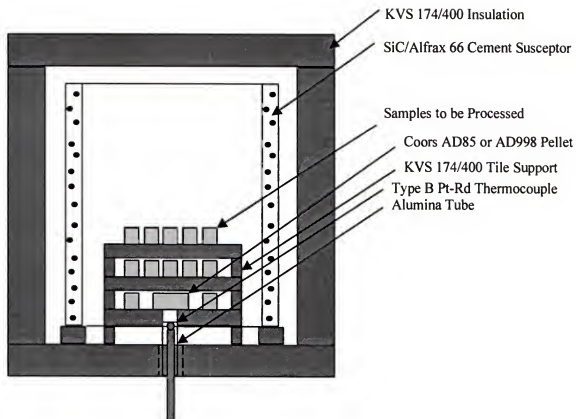


Figure 3-7. Final Assembly for Sintering Green Tiles

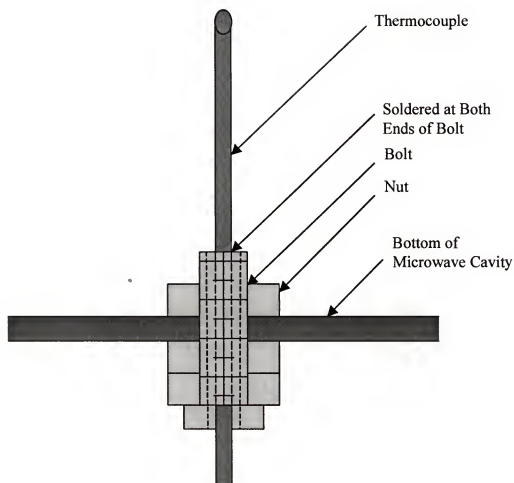
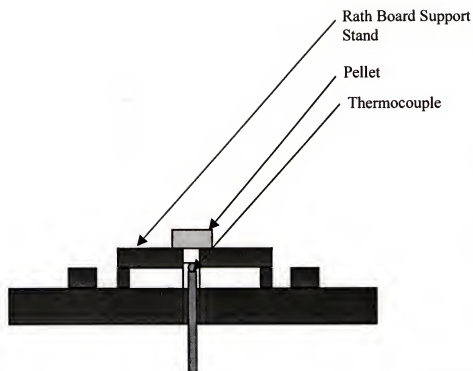


Figure 3-8. Grounding of Thermocouple Assembly

secured by two nuts. An additional nut served as a spacer for height adjustment of the thermocouple in the cavity. The use of the grounding technique helped to prevent any fluctuations of the thermocouple reading associated with the microwave field.

Before a firing run, the thermocouple was inserted through an Omegatite™ 450 thermocouple insulation tube in the bottom of the microwave (hybrid) heating assembly. The tube was made of 99.8% alumina, and was open at both ends. After insertion into the tube, the thermocouple was repositioned such that its tip was less than 0.52 cm below the bottom surface of the pellet to be sintered in the majority of sintering runs. The pellet rested on a Rath board support stand. The stand had a hole drill through its center that was slightly greater than the outer diameter of the alumina tube. The stand was aligned so that the tube would fit through its hole. The pellet was placed on the stand such that its center would align with the center of the hole. A schematic of the properly positioned tube, stand, and pellet is shown in Figure 3-9. This alignment procedure helped to insure that the temperature of the pellet would be the primary temperature read during a sintering run.

A calibration of this thermocouple with the thermocouple used to monitor the conventional sintering experiments (in Deltec furnace, mentioned in next chapter) showed that there was a maximum of $\pm 6^{\circ}\text{C}$ difference between the two thermocouples at temperatures from 1400-1600°C. During a firing run, temperature was held to within $\pm 4^{\circ}\text{C}$ of the desired soak temperature through the entire soak time.



Side View

Pellet

Thermocouple Tip



Top View

Figure 3-9. Alignment of Pellet, Stand, and Thermocouple

CHAPTER 4 MATERIALS AND METHODS

Characterization of Starting Powders

Two spray-dried powders were used to form green bodies in the experimental study. These powders included both the relatively pure Coors AD998 alumina and a less pure Coors AD85 alumina. The powders were characterized to determine the required temperature for binder burnout, their particle size distributions, their shape and structure, and their true densities.

Thermogravimetric and Differential Thermal Analysis:

Thermogravimetric analysis and differential thermal analysis was performed on the powder samples to determine the required temperature for binder burnout from the samples. The analysis was performed using a Harrop Industries Model ST-736 Simultaneous Differential Thermal Analyzer/Thermogravimetric analyzer with binder-free 99.95% pure Reynolds Metals alumina powder as a reference. The powders were set to ramp from room temperature to 1000°C at a rate on the order of 10°C/min.

Figures 4-1 and 4-2 present the results of the analysis for the Coors AD85 and Coors AD998, respectfully. In both figures, there was a large spike in the difference between the reference temperature (ΔT) and the sample temperature at approximately

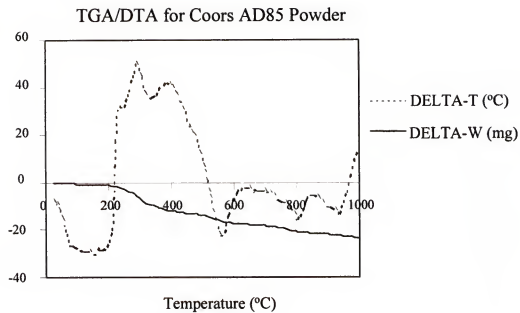


Figure 4-1. TGA/DTA Data on Coors AD85 with Added Binder

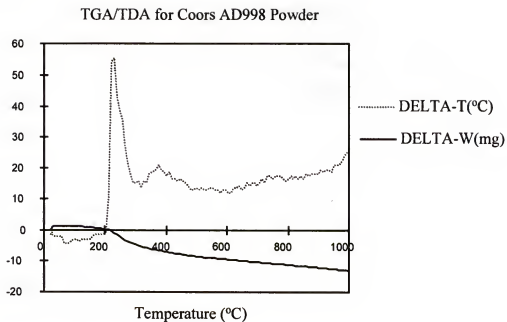


Figure 4-2. TGA/DTA Data on Coors AD998 with Added Binder

200°C, coupled with the start of a large amount of mass loss from the sample. Both were indicative of the start of binder removal from the sample. When the samples reached approximately 500°C, the majority of the binder had been removed and ΔT significantly decreased. The Coors AD85 sample, however, showed some continued variation in ΔT at temperatures above 600°C. Since the Coors AD85 powder was less pure, this variation may have been due to the softening of the glass in the powder.

Particle Size Analysis

The particle size distributions of the two powders were determined using a Coulter LS 320 particle size analyzer. A sample of each of the two powders was prepared for analysis by first removing the binder at 600°C for at least 1.5 hours. A sample of the respective powder was then suspended in deionized water and the particle size distribution (PSD) determined. The results of the analysis are presented in Figures 4-3 and 4-4.

The figures suggested that the majority of the volume of the powder consists of large particles of average size of 3.8 μm , and 152.7 μm for the Coors AD998 and AD85, respectfully. However, most of the particles found in the powder had mean sizes of 1.5 and 2 μm .

Scanning Electron Microscopy

Verification of the particle size distributions of the powders as well as further insight into their shape and microstructure was provided using scanning electron microscopy. The analysis was performed on binder-free samples of each of the two

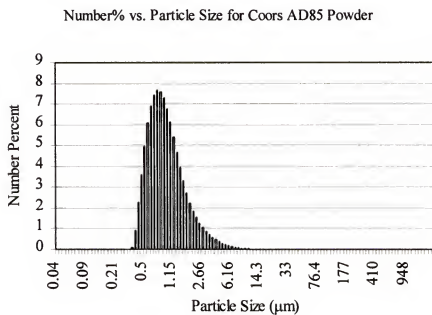
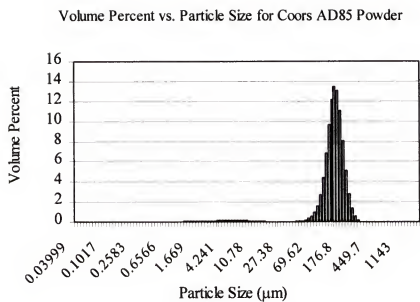
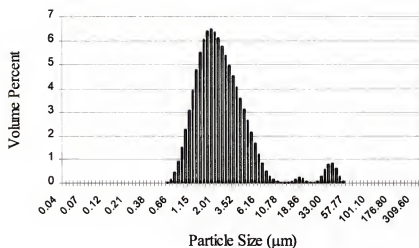


Figure 4-3. Particle Size Distribution for Coors AD85 Spray-dried Powder

Volume Percent vs. Particle Size for Coors AD998
Powder



Number Percent vs. Particle Diameter for Coors AD998 Powder

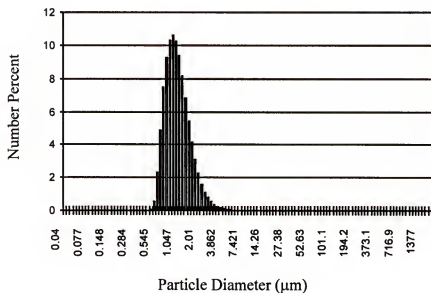


Figure 4-4. Particle Size Distribution for Coors AD998 Spray-Dried Powder

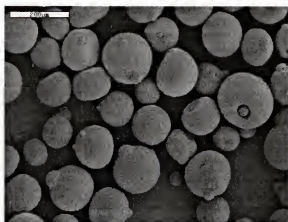
powders using a JEOL JSM-6400 scanning electron microscope. A coat of gold-palladium was applied to the samples to improve the powders' electrical conductivity. The results of the analysis have been provided in Figures 4-5 and 4-6 for magnifications of 900X, 1200X, and 10000X.

At the higher magnifications, both the Coors AD998 and Coors AD85 powders appeared to be composed of large particles with dimensions on the order of 100 μm . The Coors AD85 particles are primarily spheres, while the Coors AD998 particles are composed of both spheres and irregular shaped particles. As the magnification was increased, and close-ups of the surfaces of the particles were revealed, it appeared that the Coors AD998 particles were actually large agglomerates of smaller particles on the order of 1 μm . These large agglomerates were broken up into these smaller particles during particle size analysis, which explained the lack of the large agglomerates in the volume percent vs. particle size data.

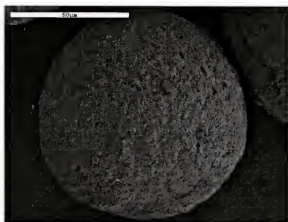
Unlike the Coors AD998 powder, the Coors AD85 did not appear to be made solely of agglomerated particles. There appeared to be a web of material binding the particles together and smoothing the surface of the larger particle. The web of material was likely a glass mixed with the alumina particles so as to promote liquid phase sintering. This glassy web bound the smaller alumina particles strongly enough so that they were not broken up during particle size analysis.

Pycnometer Density

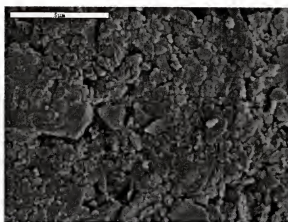
The pycnometer densities of binder-free AD85 and AD998 alumina powders were determined using a standard pycnometry technique with 0.06 ppm NaCl deionized water



900X

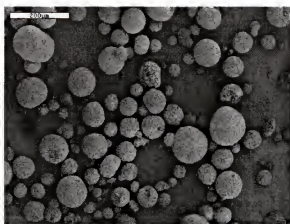


1200X



10000X

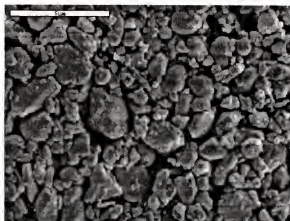
Figure 4-5. Scanning Electron Microscope Image of Coors AD85 Spray-dried Powder at Various Magnification.



900X



1200X



10000X

Figure 4-6. Scanning Electron Microscope Image of Coors AD998 Spray-dried Powder at Various Magnifications.

as wetting fluid. The principle behind this technique was that the pycnometer densities would approximate the true densities of the ceramics due to the minimal amount of closed porosity in the powders. Based on this assumption, it was then possible to substitute the pycnometer densities in for the true densities in relative density calculations on the fired ceramics.

The pycnometer density determined for the Coors AD85 powder was 3.61 g/cc. The Coors AD998 powder was assigned the pycnometer (true) density of 3.9 g/cc. This value was listed on the Coors Ceramic Company website as a typical final density for fired bodies of this material. Based on the pycnometer density for the Coors AD85 powder, the volume fraction of glass and aluminum oxide were determined using the equation

[Equ. 4-1]

$$\rho_{alumina} v_{f_{alumina}} + \rho_{glass} v_{f_{glass}} = \rho_{total}$$

where,

$\rho_{alumina}$ = density of alumina (3.98 g/cc)

ρ_{glass} = density of soda-lime-silica glass (~2.5g/cc)

$v_{f_{alumina}}$ = volume fraction of alumina in the Coors AD85 powder

$v_{f_{glass}}$ = volume fraction of glass in the powder

ρ_{total} = pycnometer density of Coors AD85 powder

The volume percentage of glass in the Coors AD85 alumina powder was determined to be 25%. This corresponded to a mass percentage of glass in the powder of 15.7%.

From the work of Kingery [King59], 30 volume percent of the shrinkage during the densification of a body with 25 volume percent of glass (liquid phase) will result from the particle rearrangement mechanism (first stage of liquid-phase sintering).

Energy Dispersive Spectroscopy (EDS)

Energy dispersive spectroscopy was performed on binder-free, carbon-coated samples of the two powders in order to qualitatively determine their composition. The analysis was performed using the JEOL JSM-6400 Scanning Microscope with an Oxford EDS analysis system. The accelerating voltage used in the analysis was 15 KeV. Data interpretation was provided by an Link Isis data interpretation program. The results of the analysis have been provided in Figure 4-7.

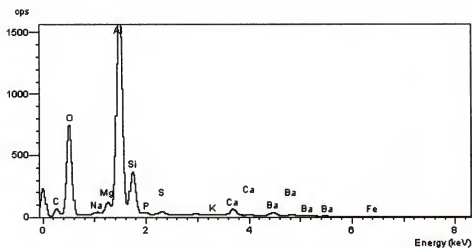
As expected, the analysis on the Coors AD85 powder (a) showed the presence of aluminum and oxygen. It also showed the presence of silicon, sodium, calcium probably from a soda-lime-silica glassy phase used to provide liquid phase sintering. The magnesium in the powder was probably from a magnesia grain growth inhibitor that was added to the powder, while iron and potassium were possibly impurities present in the glass or alumina particles.

The spectrum for the Coors AD998 powder (b) was dominated by the presence of both aluminum and oxygen. This was expected since it was a more pure alumina oxide material. Sodium and calcium, probably in the form of sodium oxide and calcium oxide, were also found in the powder. A trace of phosphorus impurity could also be detected.

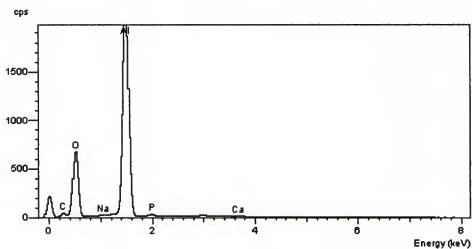
Green Body Formation

Compaction

The Coors AD85 and Coors AD998 powders were compacted to form green bodies of various shapes using stainless steel dies. Pressed shapes included bars and cylindrical pellets with footprints of 1 in² and masses of 12.7 and 15 grams, and 2" x 2"



(a)



(b)

Figure 4-7. Results of EDS Analysis on (a) Coors AD85 Spray-dried Powder and (b) Coors AD998 Spray-dried Powder

tile with masses ranging from 38 to 60 grams. These masses of starting particles were selected for green body fabrication because they would result in final fired alumina pieces having thickness of less than 10 mm (thin pieces). A light dusting of the top and bottom parts of the dies with Sumitomo AKP-15 alumina was used to prevent sticking of the bodies to the die. A hand-operated, single-end Carver Model B Uniaxial Laboratory Press applied the pressure required for compaction.

An optimal pressure for pressing the samples was determined by the pressing of Coors AD85 15 gram cylindrical pellets at progressively higher pressing pressures and measuring the pellet's resulting bulk green density. The results are shown in Figure 4-8. The results suggest that there are only small increases in bulk density above a pressing pressure of 3000 psi. Therefore 3000 psi was the pressure used to compact samples.

The green density of the Coors AD85 bodies pressed at 3000 psi was 2.12 g/cc. Based on the pycnometer density of 3.61 g/cc, this was equivalent to a relative density between 58 to 59% theoretical.

Binder Burnout

After the green body was formed, it was removed from the die and placed in a storage cabinet to await binder burnout. When it was time for binder removal, the pellet was taken from the storage chamber and placed on a bed of greater than 150 μm corundum powder (used for lubrication during sintering) that rested on a Rath, Inc. KVS 174/400 Fiberboard setter plate. The entire assembly was transferred to a Thermolyne 1400 FB box furnace and its binder removed at the conservative temperature of 600°C for

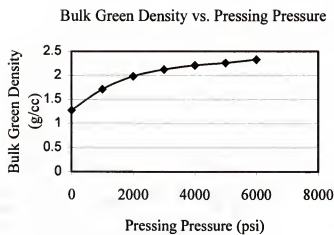


Figure 4-8. Relationship Between Bulk Green Density and Pressing Pressure for 15 g Samples of Coors AD85 Alumina.

a period of not less than 90 minutes. The assembly was then allowed to cool to room temperature before it was placed in a apparatus for firing of the pellets.

Conventional and Microwave (Hybrid) Sintering

After cooling to room temperature, the assembly was ready to be fired in either the microwave sintering assembly or a conventional furnace.

Conventional Sintering

Conventional sintering was conducted in a Deltec model DT/31/RS/12/B furnace. The furnace utilized molybdenum disilicide electrical resistance heating elements. The ramp rate was limited to about 100°C/hour, while its maximum soak temperature was about 1650°C. The interior of the cavity was able to accommodate several setter plates stacked one on top of the other.

A programmable controller was used to program the heating schedule of the furnace. Furnace temperature was measured using an alumina sheathed type-B platinum-platinum-rhodium thermocouple. The inherent accuracy of the temperature reading of the thermocouple was $\pm 6^\circ\text{C}$. However, the addition of the alumina sheath caused the thermocouple to read lower than the cavity temperature by 10-15°C. The controller provided an additional source of error of up to 15°C.

Microwave (Hybrid) Heating

Microwave (hybrid) heating was conducted using a Raytheon Radarline Model QMP 2101 B-6 microwave oven. This microwave utilized up to 8 magnetrons of 800 watts each for a maximum of 6.4 kW of 2.45 GHz microwave power. The cavity of the

microwave was multimode and had an interior volume of 5.3 ft³. Inside this cavity were 8 mode stirrers that were used to improve the uniformity of the microwave field.

The duty cycle of the microwave was controlled using a dial switch. This dial could vary the duty cycle from 0-100% during operation of the microwave.

The microwave susceptors shown in Figure 3-4 were made from a combination of Carbolon 16 GRN silicon carbide and Alfrax 66 castable alumina cement. Four different susceptors were used in this study. These include ones made from 10, 20, 30, and 60 weight percent silicon carbide.

The susceptor were made by first dry mixing the silicon carbide and Alfrax 66 alumina cement in batches of 300 to 400 grams. 0.06 ppm NaCl was added and mixed into the batch until sufficient wetting occurred. The consistency of the mixture at this point was that of a thick shake. The wetted batch was then poured into a Plexiglas mold. The mixing procedure was repeated until the entire mold was filled.

After the mold was filled, the silicon carbide/alumina cement mixture was allowed to set for at least 24 hours. The hardened susceptor was then removed from the mold and placed in a Blue M oven set at 110°C for 4-5 hours in order to dry any excess water that remained in the susceptor.

Upon completion of drying, the susceptor was transferred to the Deltec Furnace and ramped to 1500°C at the rate of 100°C/hr. The susceptors were then soaked at 1500°C for 1 hour to remove any residuals, and allowed to furnace cool.

Densification Studies

Before beginning any experimental work on Coors AD85 alumina bodies, some preliminary experiments were conducted on Coors AD998 samples to determine the firing temperature required to produce dense samples in reasonable firing times (90 minutes or less). Priority was given to research on this powder because its higher purity would likely result in fired bodies having better mechanical properties.

The drawback to firing these higher purity bodies would likely be a higher required firing temperature. If this firing temperature was too high, damage to the microwave hybrid heating chamber or conventional furnace could result. This damage would be exacerbated by the large number of firing runs that proper mechanical assessment of the samples would require.

To estimate a required firing temperature for the production of dense (95% relative density) bodies, 15 gram samples of Coors AD998 alumina were fired at temperatures at the upper range of safe operation of the microwave and the conventional furnace. The results of these preliminary experiments have been shown in Figure 4-9.

It was apparent from the results that the pellets required temperatures on the order of 1600°C in order to be fired to 95% relative density in less than 90 minutes. Temperatures near 1600°C pushed the safe limit operations of the microwave or conventional furnace. Moreover, damage to the microwave hybrid heating assembly was evident even at these firing temperatures. The damage was especially evident after several firing runs. For these reasons, further experimental work on Coors AD998 alumina was abandoned, and focus was shifted to the Coors AD85 alumina.

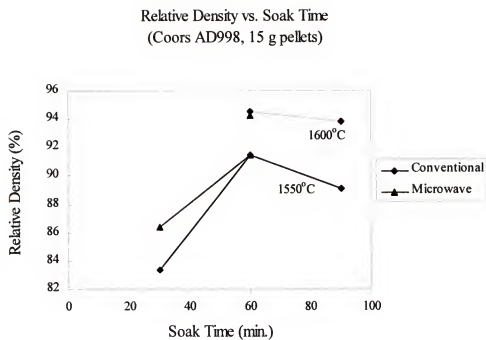


Figure 4-9. Results of Preliminary Experiments on Microwave Hybrid Heating and Conventional Firing of Coors AD998 alumina pellets. Note: Microwave Hybrid Heating was Conducted Using a 20 Weight Percent SiC Susceptor.

Before any Coors AD85 samples were sintered to full density, several densification studies were undertaken to evaluate the effects of microwave processing on the densification of 12.7 gram and 15 gram samples. Of particular interest was the densification rate in microwave processed samples compared to conventionally processed samples. There was additional interest in determining the effect of weight percent silicon carbide in the susceptor on the rate of densification in the microwave fired samples. Densification comparisons were made between microwave and conventionally processed samples fired at 1200 to 1500°C for 30 minutes.

Microwave processed samples were heated in the microwave (hybrid) assembly (Figure 3-7) by the Raytheon Radarline microwave through the application of 3200 W of 2.45 GHz microwave power. The typical heating rate to processing temperature was 35°C/min. Samples were cooled from the processing temperature at a rate of 5°C/min to 860°C, and then allowed to furnace cooled. Several susceptors composed of 10 to 60 weight percent silicon carbide were used in the experiments.

Conventionally fired samples were heated in the Deltec furnace to the processing temperature at a rate of 1.7°C/min (100°C/hr). After processing, samples were cooled at a similar rate as used in the microwave experiments. The processing schedules for both microwave and conventional heating are shown in Figure 4-10.

Two bars and one pellet of identical weights were processed in each processing run. They were arranged as shown in Figure 4-11. Archimedes principle was used to determine the densities of the processed samples.

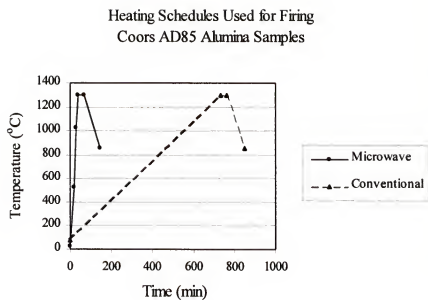


Figure 4-10. Heating Schedules for Coors AD85 Alumina Samples

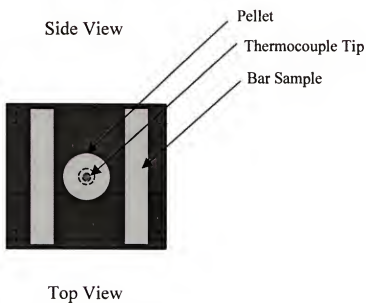
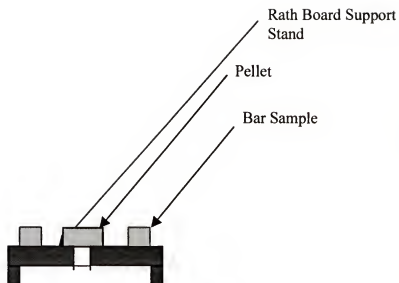


Figure 4-11. Alignment of Pellet, Stand, and Thermocouple

Batch Processing of Samples

It was necessary to densify a larger batch of samples for mechanical tests due to statistical concerns. A total of ten to twelve Coors AD85 bar samples were processed in each batch. The unfired samples were processed using schedules that would result in the fired products having relative densities of greater than 95%. Altogether, five batches were processed.

Two of the five batches were comprised of bar samples weighing 15 grams. One of these two batches was fired in the conventional furnace using a similar heating schedule as the one used in the densification studies. The second batch was fired using microwave (hybrid) heating in the Raytheon Radarline microwave oven using 3200 W of 2.45 GHz microwave radiation. The susceptor that was used contained 20 weight percent silicon carbide. Both the conventionally fired batch and microwave fired batch were processed at 1500°C for 60 minutes.

The remaining three batches were comprised of 12.7 g bars. One of these three batches was fired at 1500°C for 30 minutes in the conventional furnace. Its processing schedule was otherwise similar to that used in the densification studies. The other two batches were fired using microwave (hybrid) heating in the Raytheon Radarline microwave oven using 3200 W of 2.45 GHz microwave power. One of these two batches utilized a 20 weight percent silicon carbide susceptor and was fired at 1400°C for 30 minutes. The other utilized a 60 weight percent silicon carbide susceptor having an additional sixty weight percent silicon carbide piece to cover its top. It was fired at 1425°C for 30 minutes.

Before a batch processing run, the unfired samples were arranged on the setter plates so that no space would remain between adjacent samples. A maximum of five bar samples was processed per setter plate. A schematic of a typical processing set-up is shown in Figure 4-12.

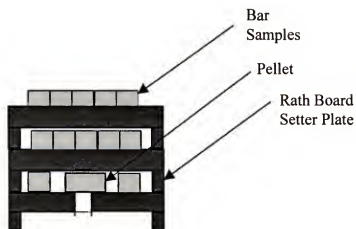
Characterization

The microwave and conventionally fired bars were characterized to determine bulk densities, microstructure, and composition.

Density Measurements

Density measurements were made on the fired Coors ceramics using Archimedes' principle. After firing and cool down, excess moisture was removed from the samples by placing them in a Blue M Stabil-Therm Constant Temperature cabinet for a minimum of 24 hours at a temperature of at least 100°C. After the excess moisture was removed, the dried bodies were weighed and transferred to a deionized water bath. The deionized water contained less than 0.06 ppm NaCl and was used to saturate all open pores in the samples. Saturation was accomplished by boiling the samples in the water bath for a period of 5 hours using a hot plate as the heat source. After boiling was completed, the samples were then allowed to cool and soak in the water bath for 24 hours. Their saturated weights in air and their suspended weights in 0.06 ppm NaCl were then measured.

Knowing the dry weights, saturated weights and suspended weights of ceramic samples, bulk densities were calculated using



Side View



Top View

Figure 4-12. Batch Processing Set-up

[Equ. 3-1]

$$\rho = \frac{\rho_w W_d}{W_A - W_s}$$

where,

ρ = bulk density of specimen (g/cc)

ρ_w = density of deionized water at room temperature (~1.0 g/cc)

W_d = dry weight of the specimen (g)

W_A = saturated weight of sample in air (g)

W_s = saturated weight of specimen in water (g)

Knowing the pycnometer density and bulk density of the specimens, relative densities were calculated using

[Equ. 3-2]

$$\rho_r = \frac{\rho}{\rho_c} * 100$$

where,

ρ_r = relative density of the specimen (%)

ρ_c = pycnometer density of the ceramic (g/cc)

The uncertainty in the relative density was determined to be +/- 0.3%. It was determined from measurement uncertainty and repeated measurements on samples

Microscopy

Scanning electron microscopy was conducted on the fired Coors AD85 pellets to help qualitatively verify trends in density and to explain differences in mechanical performance. The pellets were selected for examination because it was speculated that their smaller surface area to volume ratio would increase the risk of interior microstructure variation due to decreased temperature uniformity within the sample.

In preparation for microscopy, the pellets were sectioned in half using a Buehler Isomet Low speed saw with a Mark V model DB412 4" diameter arbor diamond blade. A cross-section from each pellet was then progressively polished on a Buehler Polimet Polisher using 180-600 grit silicon carbide polishing paper followed by 17.0, 9.5, 1.0, and 0.3 μm alumina polishing powder. The polished cross-sections were thermally etched at 1150°C for 2 hours to help bring out the grain boundaries in the samples [Zipp91]. A coat of gold-palladium was applied to the samples to improve their electrical conductivity.

The samples were examined using a JEOL JSM-6400 scanning electron microscope. In order to detect any gradients in microstructure throughout the samples, the examination was conducted on both the near surface and center of the cross sections at magnifications of 400x and 4000x. A schematic of the examination is shown in Figure 4-13.

Mechanical Property Testing

Mechanical property testing was performed concurrently to sample density and microscopy characterization. These tests included both Vicker's hardness tests and strength tests on indented samples.

Hardness Testing

Hardness testing was performed on sintered bars (relative density 95%+) of Coors AD85 using a Buehler Micromet 3 Microhardness Tester with a Vicker's hardness indentor. Test specimens included 12.7 gram bars and 15 gram bars processed using

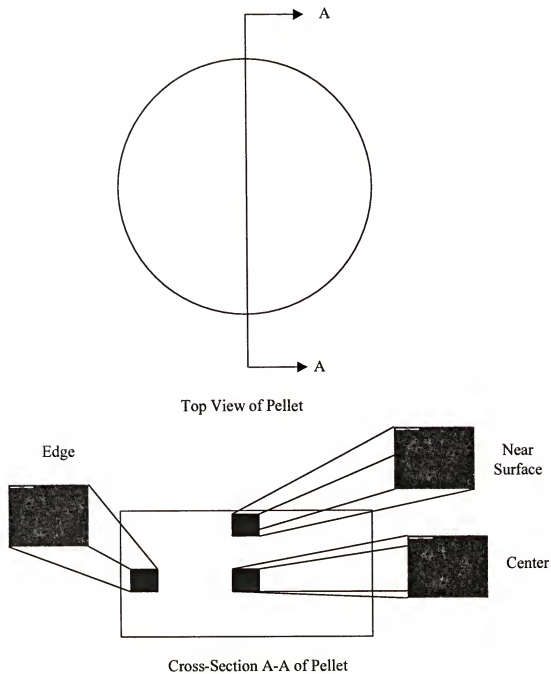


Figure 4-13. Schematic of Microstructure Analysis

microwave hybrid heating and conventional firing. The load selected for indentation was 2 kg_f, and the time of load application was 30 seconds.

The bar samples were prepared for hardness testing by polishing the test surface incrementally down using silicon carbide polishing paper (180 to 600 grit), and then fine alumina powder (17.5 µm to 1 µm) through the assistance of a Buehler Polimet polisher. After polishing, the samples were sectioned to better fit on the test stand using a Buehler Isomet Low speed saw with a Mark V model DB412 4", 12000th, ½" arbor diamond blade

The samples were marked into five equal sections using a number two pencil. Five hardness tests per section were then performed on the top surface of every bar, for a total of 25 tests per bar. After completion of testing on the top surface, further tests were performed on a cross-section of the each bar, near the middle of the bar. Five hardness tests were performed near the center of the cross-section and five tests were performed near the surface of the cross section. Schematics of both the tests on the top surfaces and cross-sections are provided in Figure 4-14. All tests were randomized on a per section basis in order to minimize potential bias.

The hardness of the bars was determined by measuring the diagonals of the pyramidal indent left on the samples. The formula used to determine the hardness was then [ASTM C 1327-96a]

$$H_v = \frac{0.0018544 F_g}{D_{avg}^2}$$

[Equ. 3-3]

where,

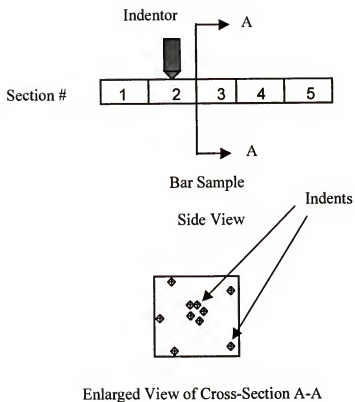


Figure 4-14. Hardness Testing on Top Surfaces and Cross-Sections of Bars

H_v = Vicker's Hardness (GPa)

F = Applied Load (Kg)

$g = 9.81 \text{ m/s}^2$

D_{avg} = Average length of two diagonals (mm)

Four-Point Flexure Testing

Four-point flexure testing of pre-indented bars was performed on the batch processed 12.7 and 15 gram bars of Coors AD85 alumina in order to assess their strength. Samples were prepared for testing by polishing the intended tensile surface in the flexural tests, and rounding its edges to minimize failure from the edges. The center of the polished surface was then indented with a Vicker's hardness indent. Of the ten samples to be tested for a given batch, four were polished on their bottom surface. The remaining six samples were polished on their top surface. The rotation of polishing provided information indicative of the uniformity of strength from top to bottom.

The 15 gram samples were polished using 120 grit silicon carbide polishing paper. Half of the samples in a given batch (two with polished bottoms, three with polished tops) were indented using a 0.5 kg_f load, while the other half was indented with a 1.0 kg_f load. These combinations of indentation load and surface finish did not promote the breaking of the samples at the indents. It was therefore necessary to polish the surfaces of the remaining samples to a finer finish and indent then using a greater indentation load.

The 12.7 gram samples were polished progressively down using 180, 240, 320, 400, and 600 grit silicon carbide polishing paper. Half the samples were indented using a 2 kg_f load, while the other half was indented using a 5 kg_f load. All indents were applied

for 30 seconds. Indented samples were stored under atmospheric conditions for several days before four-point flexure tests were conducted

The four-point flexure tests were conducted on a MTS 64205 bend fixture designed for flexural strength tests on ceramics and composites [Figure 4-15]. The bend fixture had an outer span of 40.00 mm +/- 0.10 mm and an inner span of 20.00 mm +/- 0.10 mm. Samples were placed on the fixture and carefully aligned in the z-direction to prevent excess shear on the samples.

The load for the flexure tests was applied using a Model 1125 Intron Test Machine with a 5000 lb load cell. The tests were conducted at a strain rate on the order of 1×10^{-4} per sec according to MIL-STD-1942 and under a full scale of load of 2000 lbs for a total uncertainty of +/- 10 lb. The samples were preloaded to 20 lbs before flexure testing to assist in sample settling on the fixture. The breaking stress for the samples was calculated using [MIL-STD-1942 (MR)]

[Equ. 3-4]

$$S = \frac{3PL}{4bd^2}$$

where,

- S = breaking stress (psi)
- P = load required to break the sample (lb_f)
- L = length of outer span of test fixture (in)
- t = thickness of bar (in)
- b = width of bar (in)

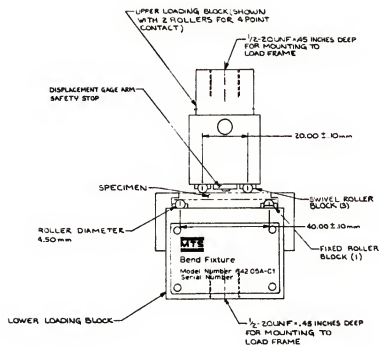


Figure 4-15. Schematic of Four-point Flexure Test Fixture

Uncertainty Analysis

The uncertainty in the density, hardness, and modulus of rupture of the alumina samples was determined by a root-sum-square of the bias limit and the precision limit of the experimental results [Cole89]. The equation used to determine uncertainties in the data had the form

[Equ. 3-5]

$$U_{RSS} = [B_r^2 + P_r^2]^{1/2}$$

where,

U_{RSS} = uncertainty in the result determined by the root-sum-square technique

B_r = bias limit of the experimental result

P_r = precision limit of the experimental result

The bias limit was based on measurement error in the experimental variables that are used to determine the experimental result. For an experimental result, r , which is a function of variables X , Y , and Z ,

$$r = r(X, Y, Z)$$

The bias limit has the form of

[Equ. 3-6]

$$B_r = \left[\left(\frac{\partial}{\partial X} U_X \right)^2 + \left(\frac{\partial}{\partial Y} U_Y \right)^2 + \left(\frac{\partial}{\partial Z} U_Z \right)^2 \right]^{1/2}$$

where,

$U_{X,Y \text{ or } Z}$ = uncertainty in variables X , Y , or Z

Uncertainty in variables X , Y and Z usually stem from potential errors associated with readout, or experimental bias.

The precision limit was determined from the results of repeated experiments. Assuming a Gaussian distribution for the repeated experiments, the precision limit was a 95 percent confidence interval of the mean of the experimental results. Mathematically, the precision limit was determined using

[Equ. 3-7]

$$Pr = \frac{tS_x}{\sqrt{N}}$$

where,

t = student-t variable for 95% confidence and N-1 degrees of freedom

S_x = estimate of the standard deviation based on N experiments

N = number of experiments

CHAPTER 5

RESULTS

The results of the study were divided into two sections. The first section has focused on the results of the densification experiments, while the second has focused mechanical property testing. Both sections have been augmented with microstructure analysis to support trends in the data and provide further information on microstructure development using the two firing techniques.

Densification

The results of the densification studies have been presented for both the 12.7 gram samples and the 15 gram samples of Coors AD85 alumina powder. The results for the 12.7 g samples have been presented in Figure 5-1 and Table 5-1. Figure 5-1 plotted the relative density vs. processing temperature for conventional firing and microwave hybrid heating using alumina cement susceptors composed of 10 to 30 weight percent silicon carbide, and one of 60 weight percent silicon carbide with an additional 60 weight percent silicon carbide top. The data in this figure has also been tabulated in Table 5-1. The relative densities plotted in the figure and expressed in Table 5-1 were averages of two bar samples and one pellet sample.

For identical firing temperatures, the results suggested that microwave hybrid firing did increase the amount of densification as compared to conventional firing of the Coors AD85 alumina samples. The percentage increase in densification over

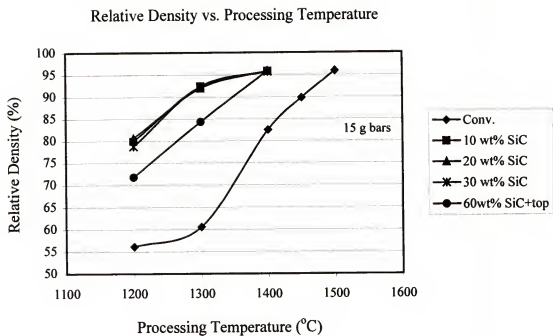


Figure 5-1. Densification of Microwave and Conventionally Fired 12.7 g AD85 Alumina Samples (Average of Pellet + 2 Bars).

Table 5-1. Relative Density vs. Processing Temperature for Microwave and Conventionally Processed 12.7 g Coors AD85 Samples

Firing Temperature (°C)	Conventional	10 wt% SiC Susceptor	20 wt% SiC Susceptor	30 wt% SiC Susceptor	60 wt% SiC Susceptor + Top
1200	56.2	79.9	80.7	78.7	71.8
1300	60.6	92.4	92.0	92.4	84.4
1400	82.5	95.8	95.9	95.7	95.9

conventional firing by the different cases of microwave hybrid heating has been presented in Table 5-2.

Microwave hybrid heating using the 10 to 30 weight percent silicon carbide susceptors resulted in similar amounts of densification increase over conventional firing. The average increase in densification over conventional firing for microwave hybrid heating using these susceptors was 41.9 %, 52.3 %, and 16.1 % for processing temperatures of 1200, 1300, 1400°C, respectfully. Microwave hybrid heating using the 60 weight percent silicon carbide susceptor with an additional 60 weight percent silicon carbide top showed less enhancement of densification compared to the other cases of microwave hybrid heating. Microwave hybrid heating using this susceptor resulted in an increase in densification over conventional firing of only 27.8 % and 39.3 % for firing temperatures of 1200 and 1300°C, respectfully. However, repeats of the latter experiment at 1200°C resulted in densification in the Coors AD85 samples that was not as dramatically different than that produced using the 10 to 30 weight percent silicon carbide susceptors. Two repeated runs resulted in samples with relative densities of 78.4 % and 76.9 %. These values of relative density corresponded to an increase in densification over conventional firing of 39.5% and 35.1 %, respectfully.

Another interesting result from the experiments on the 12.7 gram samples involved the firing temperature required for the samples to reach ~95% relative density. It was evident from the trends in the data that it would require a firing temperature of 1500°C to fire the conventional samples to ~95% relative density in 30 minutes. All microwave hybrid heated samples were fired to ~95% relative density using a firing temperature of only 1400°C.

Table 5-2. Percentage Increase in Densification by Microwave Firing as Compared to Conventional Firing.

Firing Temperature (°C)	10 wt% SiC Susceptor	20 wt% SiC Susceptor	30 wt% SiC susceptor	60 wt% SiC Susceptor + top
1200	42.2 %	43.6 %	40.0 %	27.8 %
1300	52.5 %	51.8 %	52.5 %	39.3 %
1400	16.1 %	16.2 %	16.0 %	16.2 %

The results of the densification experiments on 15 gram Coors AD85 samples were similar to those of the 12.7 gram samples. These results have been presented in Figure 5-2 and Table 5-3. The results compare the densification of microwave hybrid heated (20 weight percent silicon carbide susceptor) 15 gram bars of Coors AD85 alumina to identical AD85 bars fired in a conventional furnace. The firing time for all cases was 30 minutes.

It was evident from the results that microwave hybrid heating again produced greater densification in the bars for a given firing temperature. The microwave hybrid heated technique required a processing temperature of only 1300°C to produce bars with a relative density of ~95%. The conventional technique required a firing temperature of 1500°C to produce bars with a similar density. This reduction in the required firing temperature was somewhat greater than that witnessed in the 12.7 gram samples.

An investigation of the microstructure development of the fired samples supported the trends in bulk density seen in the previous figures. The microstructure development in the center and the near surface regions of a 12.7 gram conventionally fired pellet and two 12.7 gram microwave fired pellets has been provided in Figures 5-3 to 5-6. The microstructure development at magnifications of 400X has been presented in Figures 5-3 and 5-4, while the development at magnifications of 4000X has been presented in Figures 5-5 and 5-6. These particular pellets were examined because of their difference in bulk density at the different firing temperatures.

It was evident that densification occurred at a similar rate in both the center and near surface regions of pellets, regardless of technique used to fire the samples. It was also evident from the figures that the densification began at a lower temperature in the

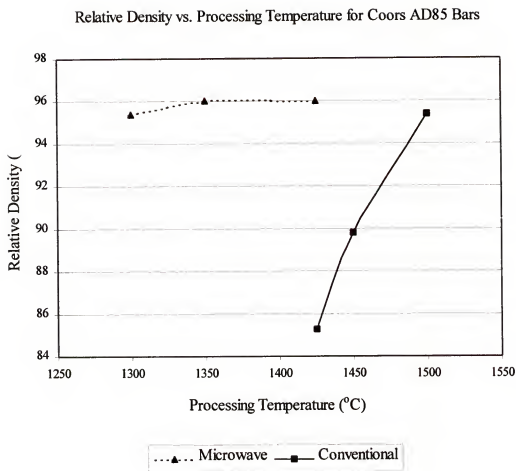
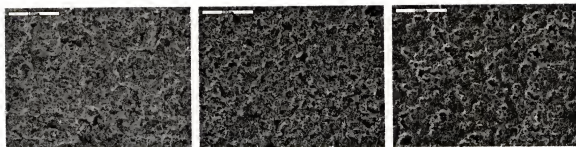


Figure 5-2. Densification of Microwave and Conventionally Fired 15 g AD85 Alumina Bars

Table 5-3. Relative Density vs. Processing Temperature for Microwave and Conventionally Processed 15.0 g Coors AD85 Bars

Temperature (°C)	Conventional	Microwave
1300	-	95.4
1350	-	96
1425	85.3	96
1450	89.8	-
1500	95.4	-

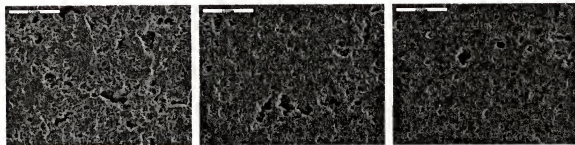


1200°C

1300°C

1400°C

CONVENTIONAL

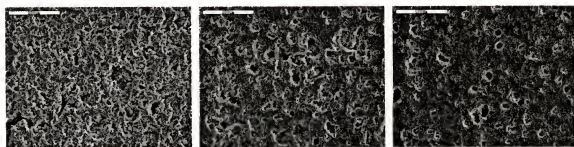


1200°C

1300°C

1400°C

20 wt% SUSCEPTOR



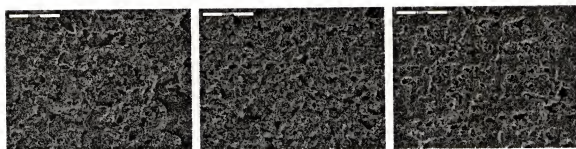
1200°C

1300°C

1400°C

60 wt% SUSCEPTOR
+ TOP

Figure 5-3. SEM Images of the Center of Conventional and Microwave Fired Pellets at 400X. Note: Scale Shown in Figures is 100 μ m.

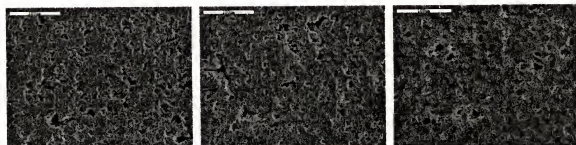


1200°C

1300°C

1400°C

CONVENTIONAL

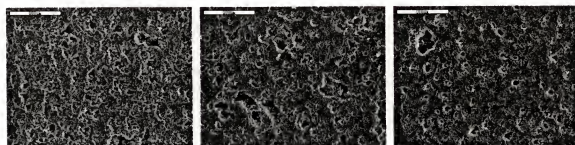


1200°C

1300°C

1400°C

20 wt% SUSCEPTOR



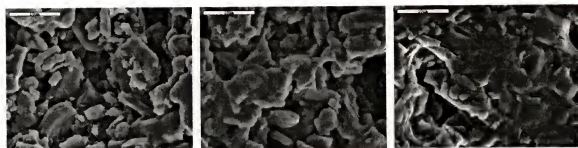
1200°C

1300°C

1400°C

60 wt% SUSCEPTOR
+ TOP

Figure 5-4. SEM Images of the Near Surface of Conventional and Microwave Fired Pellets at 400X. Note: Scale Shown in Figures is 100 μm .

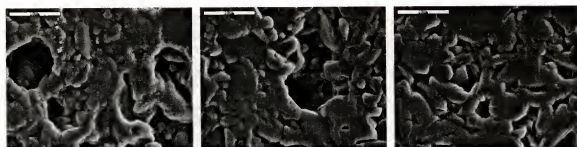


1200°C

1300°C

1400°C

CONVENTIONAL

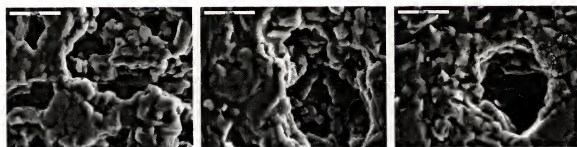


1200°C

1300°C

1400°C

20 wt% SUSCEPTOR



1200°C

1300°C

1400°C

60 wt% SUSCEPTOR
+ TOP

Figure 5-5. SEM Images of the Center of Conventional and Microwave Fired Pellets at 4000X. Note: Scale Shown in Figures is 10 μ m.

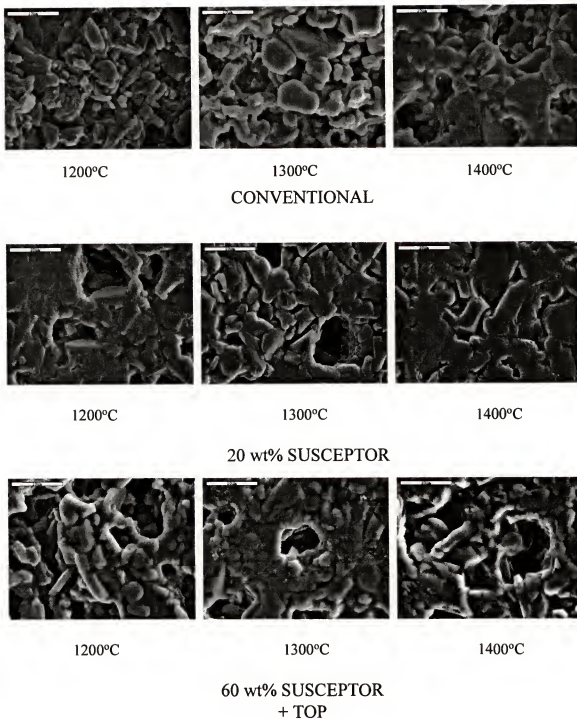


Figure 5-6. SEM Images of the Near Surface of Conventional and Microwave Fired Pellets at 4000X. Note: Scale Shown in Figures is 10 μm.

microwave hybrid heated samples than the conventionally fired sample. The level of densification in the conventional sample fired at 1400°C was about the same as that in the microwave fired sample (20 weight percent SiC susceptor) at 1200°C.

For firing temperatures of 1200 and 1300°C, the pellet fired in the microwave using the 20 weight SiC susceptor was more dense than the pellet fired in the microwave using the 60 weight SiC susceptor with an additional top.

Mechanical Testing

In order to compare the mechanical properties of the bars fired using conventional and microwave hybrid firing, mechanical testing in the form of Vicker's hardness tests and indented strength tests was performed on conventional and microwave fired 12.7 g and 15 g Coors AD85 alumina bars having ~95-96% relative density. The processing temperature and time for the bars used in these tests has been provided in Table 5-4. As noted in the table, the processing temperature for the microwave fired bars was between 75 to 200°C less than the processing temperature used to fire the conventional bars.

Hardness Testing

The results of the hardness testing on the top surface and the cross-sections of the microwave fired and conventionally fired bars have been summarized in graphical and tabular form to provide insight into the trends of the data.

Figures 5-7 and 5-8 and Table 5-5 summarized the results of the average Vicker's hardness across the top surface of the Coors AD85 bars. The results of the testing on the 12.7 gram and 15 gram bars have been presented graphically in Figure 5-7 and Figure 5-

Table 5-4. Processing Schedules for Bars Studied in Hardness and Indented Strength Testing

Sample	Processing Schedule		Relative Density (%)
	Temperature ($^{\circ}\text{C}$)	Time (min)	
Conventional (12.7 g)	1500	30	95.9
10 wt% SiC (12.7 g)	1400	30	95.7
20 wt% SiC (12.7 g)	1400	30	96.0
30 wt% SiC (12.7 g)	1400	30	95.6
60 wt% SiC + top (12.7 g)	1425	30	95.5
Conventional (15 g)	1500	30	95.4
20 wt% SiC (15 g)	1300	30	95.4

Avg. Vicker's Hardness for Each Section of 12.7 g Coors AD85
Alumina Bars
(Top Surface)

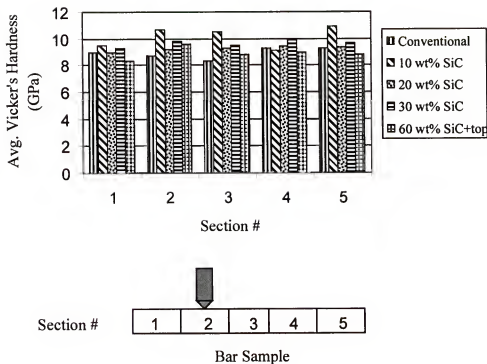


Figure 5-7. Average Hardness across Top Surface of 12.7 g Coors AD85 Alumina Bars. The Average Hardness Values Reflect the Average of 5 Measured Points within Each Section.

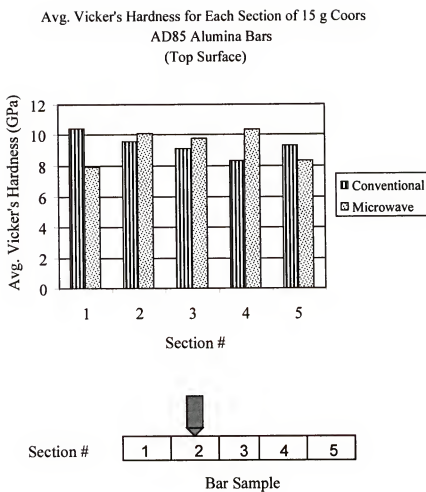


Figure 5-8. Average Hardness across Top Surface of 15 g Coors AD85 Alumina Bars. The Average Hardness Values Reflect the Average of 5 Measured Points within Each Section.

Table 5-5. Average Hardness across Top Surface of Bars

Sample	Range* of Section Average Hardness (GPa)	Range* of Uncertainty in Section Average Hardness (GPa)
Conventional (12.7 g)	8.3 to 9.3	+/- 0.6 to +/-1.7
10 wt% SiC (12.7 g)	9.1 to 10.9	+/- 0.9 to +/- 1.6
20 wt% SiC (12.7 g)	9.0 to 9.4	+/- 0.5 to +/-1.9
30 wt% SiC (12.7 g)	9.3 to 10.0	+/- 0.5 to +/- 1.0
60 wt% SiC + top (12.7 g)	8.4 to 10.4	+/- 0.8 to +/- 1.2
Conventional (15 g)	8.3 to 10.4	+/- 0.9 to +/-1.8
20 wt% SiC (15 g)	7.9 to 10.4	+/- 0.4 to +/-2.0

*Each bar was divided into five equal sections over which measurements were performed. The average hardness values reflect the average of five measured points within each section. The uncertainty values apply to the averaged hardness measurements.

8, respectfully. A more quantitative summary of these hardness results have been presented in Table 5-5.

It appeared from the figures that there was some variability in the average Vicker's hardness across the top surface of the bars, and between equivalent sections of different bars. The average hardness of the sections ranged from 8.3 to 10.9 GPa. However, uncertainties associated with the 95% confidence interval of the mean hardness values and the measurement technique were large enough to prohibit any differentiation between the average hardness of the sections. The average top surface hardness of all sections of the bars could therefore be viewed as equivalent to one another.

The hardness results on the sections of the bars were grouped together to provide average hardness values for the entire top surface of each bar. The average hardness of the top surface of any given bar was therefore based on 25 measurements (5 per section x 5 sections). The results are presented in Figures 5-9 and 5-10, and in Table 5-6.

The average Vicker's hardness for the top surface of the bars ranged from 8.9 to 10.1 GPa, with total uncertainties ranging from ± 0.3 to ± 0.6 GPa. There were a few cases where there were no overlaps in the uncertainty bands of the average hardness measurements. However, there were enough possible judgement errors in the determination of measurement uncertainty to prohibit differentiation between average hardness values of any of the bars. It was therefore concluded that the average top surface hardness was similar for all bars tested.

In order to characterize the interior hardness of the samples, additional hardness measurements were performed on the cross-sections of the bars. The results of the hardness testing on the cross-sections of the bars have been summarized in Table 5-7.

Avg. Vicker's Hardness for 12.7 g Coors AD85 Alumina
Bars
(Top Surface)

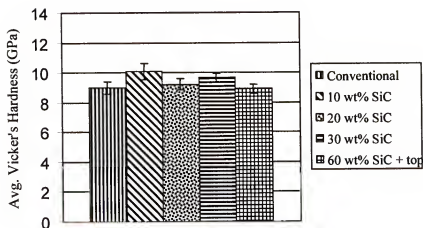


Figure 5-9. Average Hardness of Top Surface of 12.7 g Coors AD85 Alumina Bars. Average Hardness Values Reflect the Average of the 25 Measured Points across the Samples (5 measurements in each of the five sections).

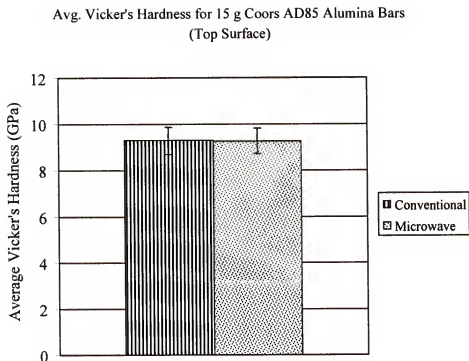


Figure 5-10. Average Hardness of Top Surface of 15 g Coors AD85 Bars. Average Hardness Values Reflect the Average of the 25 Measured Points across the Samples (5 measurements in each of the five sections).

Table 5-6. Average Hardness of Top Surface of Bars

Sample	Average Hardness* (Gpa)	Uncertainty (GPa)
Conventional (12.7 g)	9.0	+/- 0.4
10 wt% SiC (12.7 g)	10.1	+/- 0.5
20 wt% SiC (12.7 g)	9.2	+/- 0.4
30 wt% SiC (12.7 g)	9.6	+/- 0.3
60 wt% SiC + top (12.7 g)	8.9	+/- 0.3
Conventional (15 g)	9.3	+/- 0.6
20 wt% SiC (15 g)	9.3	+/- 0.5

*Average hardness values reflect the average of the 25 measured points across the samples (5 measurements in each of the five sections).

Table 5-7. Interior and Near Surface Hardness for Microwave and Conventionally Fired 12.7 gram and 15 gram Coors AD85 Bars.

Firing Technique	Interior		Near Surface	
	Average Hardness* (GPa)	Uncertainty (GPa)	Average Hardness* (GPa)	Uncertainty (GPa)
Conventional (12.7 g)	9.4	+/-0.3	8.3	+/-2.6
10 wt% SiC (12.7 g)	7.9	+/-2.5	7.6	+/-1.6
20 wt% SiC (12.7 g)	9.4	+/-1.2	9.2	+/-1.1
30 wt% SiC (12.7 g)	9.4	+/-1.0	10.2	+/-1.2
60 wt% SiC + top (12.7 g)	9.2	+/-0.6	9.0	+/-1.7
Conventional (15 g)	9.6	+/-2.5	8.7	+/-1.2
20 wt% SiC (15 g)	8.2	+/-1.4	8.6	+/-1.6

*The average hardness values reflect the average of five measured points within each region. The uncertainty values apply to the averaged hardness measurements.

The average Vicker's hardness ranged from about 7.9 to 9.6 GPa in the interior of the cross-sections, and from about 7.6 to 10.2 GPa near the edge of the cross-section. The total uncertainty ranged from about 0.3 to 2.6 GPa.

It was not possible to determine whether there was any significant difference in the average hardness across the cross-section of a given bar or between the cross-sections of different bars. The uncertainty levels were large enough so that all bars appeared to have similar cross-sectional hardness.

Flexure Testing

The results of the flexure tests on the 15 gram Coors AD85 samples fired at 1500°C for 60 minutes have been presented in Figure 5-11. Because of the combination of light indentation load (0.5 and 1.0 Kg) and limited polishing (120 grit) that was applied to these samples, the vast majority of the samples did not fail at the indent. The results have therefore been reported in terms of breaking stress.

The average strengths of these microwave fired and conventionally fired 15 gram bars were 178 and 183 MPa, respectfully. The uncertainty in the strength values was +/- 17 MPa for the microwave fired bars, and +/-12 MPa for the conventionally heated bars. Within the estimated uncertainty of the experiment, the strength of the microwave and conventionally fired Coors AD85 bars were similar.

The results of the flexure testing on the indented 12.7 gram bars of Coors AD85 alumina have been provided in Figure 5-12, and in Table 5-8. The slope of the lines in this logarithmic plot were used to verify how close the data was to the ideal slope of -0.33 (-1/3) predicted for this kind of fracture toughness data. In every case reported, failure occurred from the indent.

Four-point Bend Strength of Microwave and
Conventionally Fired 15 gram Coors AD85 Bars

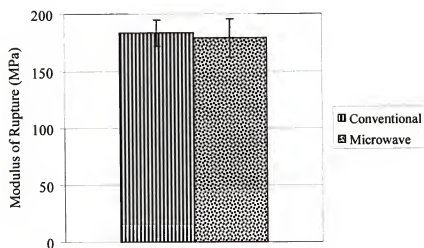


Figure 5-11. Results of Strength Testing on 15 gram Coors AD85 Bars

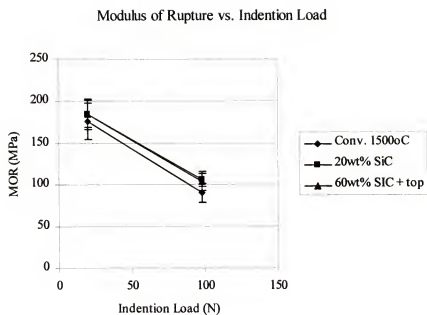


Figure 5-12. Log-log Plot of the Breaking Stress vs. Indentation Load for Coors AD85 Alumina Fired Conventionally and by Microwave Hybrid Heating.

Table 5-8. Results of the Indented Strength Tests

Firing Technique	2 Kg Indention Load		10 Kg Indention Load	
	Breaking Stress (MPa)	Uncertainty (MPa)	Breaking Stress (MPa)	Uncertainty (Mpa)
conventional	176	+/-21	90	+/-10
20 wt% SiC Susceptor	185	+/-15	107	+/-9
60 wt% SiC Susceptor w/Top	185	+/-18	103	+/-10

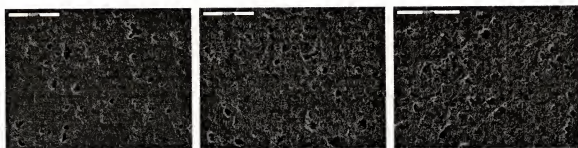
The bars indented with a 2 kg load had a mean strength that ranged from about 176 to 185 MPa. This mean strength of the bars was reduced by about 40% when the bars were indented with a 10 kg load. The mean strength values of the bars indented using this load ranged from about 90 to 107 MPa. The uncertainty associated with the data ranged from 8.0 to 12.1 %. The slopes of the lines in the logarithmic plot of the data were -0.34, -0.36, and -0.42, respectively. Within the uncertainty of the data, the slopes were in good agreement with the ideal value of -0.33 (-1/3).

Due to the level of uncertainty associated with these strength values, it was not possible to discriminate between the performance of bars produced with different firing techniques. Therefore, it was concluded that microwave hybrid heating produced bars with similar indented strength performance as conventional firing of the bars.

Final Microstructure

Images of the final microstructure of the microwave hybrid heated and conventionally fired pellets have been provided in Figures 5-13 to 5-14. The analysis done at magnifications of 400X has been presented in Figure 5-13. Figure 5-14 has presented the analysis done at a magnification of 4000X.. All three samples pictured had relative density (Archimedes principle) of ~95%.

It was evident from both figures that there was little variation in microstructure from region to region within a given sample. At magnification of 400X, all three samples appeared to have a similar microstructure. This microstructure was characterized by

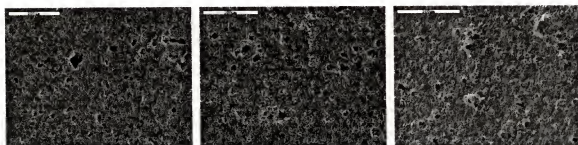


Center

Surface

Edge

CONVENTIONAL (1500°C, 30 min.)

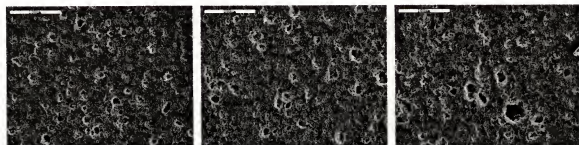


Center

Surface

Edge

20 wt% SUSCEPTOR (1400°C, 30 min.)



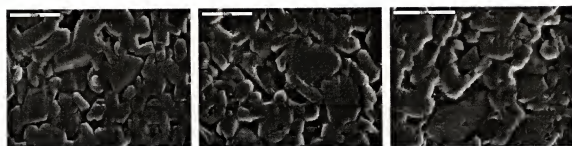
Center

Surface

Edge

60 wt% SUSCEPTOR
+ TOP (1425°C, 30 min.)

Figure 5-13. SEM Images of the Interior of Conventional and Microwave Fired Pellets at 400X. Note: Scale Shown in Figures is 100 μ m.

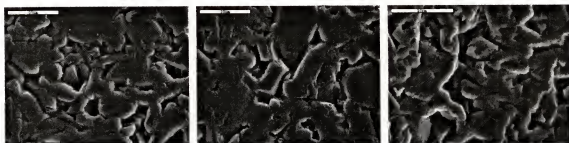


Center

Surface

Edge

CONVENTIONAL (1500°C, 30 min.)

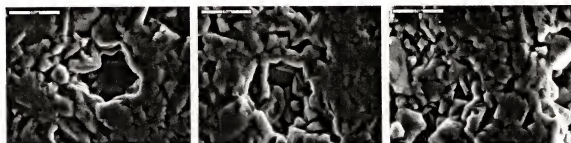


Center

Surface

Edge

20 wt% SUSCEPTOR (1400°C, 30 min.)



Center

Surface

Edge

60 wt% SUSCEPTOR
+ TOP (1425°C, 30 min.)

Figure 5-14. SEM Images of the Interior of Conventional and Microwave Fired Pellets at 4000X. Note: Scale Shown in Figures is 10 μ m.

primarily dense structure dotted with pores having diameters ranging in size from 10 to 40 μm .

At the finer magnification, some differences were apparent between the samples. While the conventionally fired pellet and one of the microwave hybrid heated pellets (20 weight percent SiC susceptor) were similar in final grain size (on the order of 10 μm), the other microwave hybrid heated sample had a grain size that was noticeably smaller. This suggested that the latter microwave hybrid heated sample had a less developed microstructure than the other two pellets, and required firing at higher temperature for a longer time to have the same final grain size as the other two cases.

Summary

It was possible to produce Coors AD85 bars with 95%+ relative density by microwave hybrid heating using firing temperatures from 75 to 200°C less than those required for conventional firing. Evidence of this increase in densification has been supported by microstructure analysis on some fired samples.

When compared at the same relative density (i.e. ~95%), microwave hybrid heated bars had similar mechanical performance as those produced conventionally at the higher required firing temperature. The microstructures of these dense, microwave hybrid heated samples were similar to those produced using conventional firing.

CHAPTER 6 DISCUSSION

Temperature Measurements

One of the most critical issues in the study was the accuracy of temperature measurement during the microwave hybrid heating experiments. The primary causes of uncertainty in the temperature measurement stem from the necessities of placing the thermocouple inside the microwave field, and avoidance of direct contact of the thermocouple with the sample.

To avoid possible interaction with the microwave field, the thermocouple used in this experiment was shielded from the microwave field by a platinum-rhodium sheath, and well grounded to the bottom of the microwave cavity. A study by Grellinger and Janney [Grel94] on temperature measurement in the microwave field suggested that these precautions would help ensure the accuracy of temperature measurement by a thermocouple. When these precautions were observed, the temperature read by the thermocouple agreed to within $\pm 20^{\circ}\text{C}$ of two other measurement devices (2-color infrared pyrometer, and optical fiber probe) that were insensitive to the microwave field.

A second area of concern was the lack of direct contact between the thermocouple and the surface of the sample. It was necessary to avoid direct contact of the thermocouple with the sample due to corrosion concerns. This lack of direct contact had the potential of causing the thermocouple to read a temperature lower than the actual

surface temperature of the sample. To ensure that this was not an issue, comparisons have been made between the heating rates of samples for varying thermocouple depths below the sample. The results of this comparison are found in Figure 6-1 and 6-2.

Since the heating rates were almost identical in each case, it was concluded that the temperature read by the thermocouple was insensitive to thermocouple distance from the sample for the range of distances used in this study. This result, coupled with the proximity of the thermocouple to the sample, verified that the thermocouple was indeed accurately measuring the temperature of the sample surface.

Microwave Penetration

Another critical issue in the study was whether microwaves would penetrate through the susceptors and impinge on the sample. If the depth of penetration of the microwaves into the susceptor was too small, then all of the microwave power incident on the susceptor would be absorbed. In this case, the sample would be heated only by radiant and conductive heat transfer from the susceptor.

The experimental technique ensured that microwave energy would reach the samples through careful selection of the susceptor material, and design of susceptors with open tops. The depths of penetration of the microwaves into alumina cement susceptors composed of varying weight percent silicon carbide content are shown in Figure 6-3. The results were created from Equation 2-5 from the data of [Cozz95] (Figures 2-27 and 2-28) and [Batt95]. Since no dielectric data was available for the 60 weight percent silicon carbide susceptor, an estimate of the dielectric properties of this susceptor was determined using the dielectric data for the alumina cement susceptor (0% SiC) (Figures

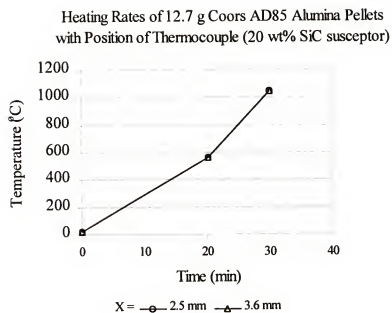


Figure 6-1. Heating rates of 12.7 g Coors AD85 Alumina Pellets with the Thermocouple Positioned at two Depths below the Bottom Surface of the Pellet.

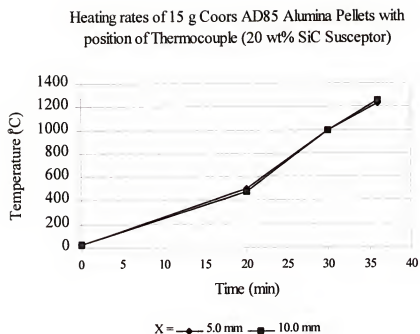


Figure 6-2. Heating rates of 15 g Coors AD85 Alumina Pellets with the Thermocouple Positioned at two Depths below the Bottom Surface of the Pellet.

Depth of Penetration into Alumina Cement/Silicon Carbide
Susceptors vs. Temperature

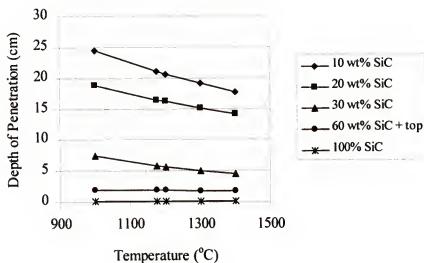


Figure 6-3. Estimated Depth of Penetration into Various Alumina Cement/Silicon Carbide Susceptors [Adapted from Cozz96, and Batt95].

2-27 and 2-28), 100% SiC data [Batt95], and the Maxwell dielectric mixture model [Moor93]

(Equ. 6-1)

$$\kappa' = \frac{v_m \kappa'_m (0.667 + \kappa'_d / 3 \kappa'_m) + v_d \kappa'_d}{[v_m (0.667 + \kappa'_d / 3 \kappa'_m) + v_d]}$$

where,

κ' = dielectric property of interest (ϵ' or $\tan \delta$)

$v_{m,d}$ = volume fraction of the matrix phase (Alfrax 66 alumina cement) or dispersed phase (silicon carbide particle)

$\kappa'_{d,m}$ = dielectric property of interest for the matrix or dispersed phase

At 1400°C, the depth of penetration ranged from about 17.7 cm for a 10 weight percent SiC susceptor to 0.14 cm for a 100 weight percent SiC susceptor. The depth of penetration of the microwaves through the 60 wt% susceptor at this temperature was estimated to be 1.8 cm.

From the depth of penetration data, it was possible to quantify microwave power that penetrated the susceptors and was available for heating the Coors AD85 alumina samples. The results of this analysis have been presented in Figure 6-4. The results have been based on susceptors with a one centimeter wall thickness (the wall thickness used in the current study). The percent power absorbed was determined by

(Equ. 6-2)

$$P_a = \frac{t_w}{DOP} \left(1 - \frac{1}{e}\right) * 100$$

where,

P_a = percent of incident microwave power absorbed by susceptor

t_w = the wall thickness of the susceptor (one centimeter)

DOP = Depth of Penetration (from Figure 6-3)

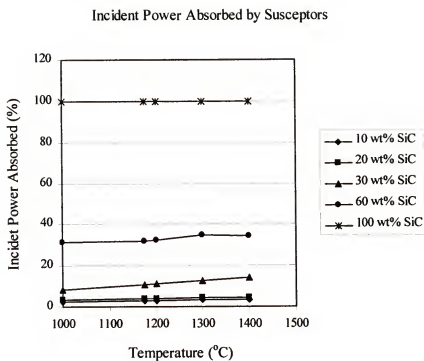


Figure 6-4. Estimated Incident Power Absorbed by One Centimeter Thick Susceptors [adapted from Cozz96 and Batt95].

The estimated power absorbed by the susceptors at 1400°C ranged from about 3.6 % to 14.1% for the 10 weight percent SiC susceptor and the 30 weight percent susceptor, respectively. This meant that from 85.9 % to 96.4% of the incident microwave energy was penetrating through the susceptors and was available to interact with the Coors AD85 samples. For an incident power of 3200 W, this translated into a power available for sample interaction of 2750 W to 3100 W. With these large amounts power available for microwave-sample interaction, it was reasonable to expect similarity between the densification of the samples using the 10 to 30 weight percent SiC susceptors. The partitioning of energy between the infrared/conductive heat transfer from the susceptors and energy of microwaves was also similar for these three susceptors.

The 60 weight percent susceptor absorbed more power than the other three susceptors used in this study. At 1400°C, it has been estimated that this susceptor absorbed about 34% of the microwave radiation that impinged upon it. This meant that 66%, or 2100 W was available for direct microwave interaction with the sample. Consequently, more of the microwave energy was transformed into infrared/conductive heat, and less microwave power was available for direct interaction with the sample. The results of the densification studies suggested that the decrease in the available microwave power for direct sample interaction was large enough to begin to differentiate it from the other microwave hybrid heated cases.

Wroe [Wroe96], in a study on microwave assisted sintering of partially stabilized zirconia at 2.45 GHz found similar decreases in the densification enhancement as the applied microwave power available for sample interaction was reduced. Some results from this study have been presented in Figure 6-5. The linear shrinkage rate at 67%

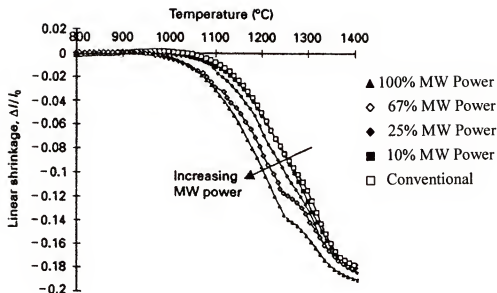


Figure 6-5. Normalized Linear Shrinkage rate of Zirconia Plotted as a Function of Sintering Temperatures for a Number of Microwave Powers. The Sintering Enhancement Increases with increasing Microwave Power [Wroe96].

applied power was less than that for 100% applied power. However, the linear shrinkage rate at all levels of applied microwave power was higher than for conventional radiant heating only. This enhancement of densification in the presence of the microwave field was attributed to increased densification-related diffusion.

Potential Causes for Enhanced Densification with Microwave Hybrid Heating

There were several possible causes for the enhanced densification provided by microwave hybrid firing relative to conventional firing of the Coors AD85 alumina samples processed at identical firing temperatures. Some of these potential causes could be explained in terms of thermal effects, while others could only be explained in terms of non-thermal effects. It was probable that the enhancements of densification were due to both thermal and non-thermal effects.

Heating Rate

It was unlikely that the differences in densification between the microwave hybrid heated and conventionally fired bodies were due to differences in heating rates at which the samples were ramped to firing temperatures. Though there were large differences in the heating rates between the two cases ($\sim 35^{\circ}\text{C}/\text{min}$ for MHH and $\sim 1.6^{\circ}\text{C}/\text{min}$ for conventional firing), it did not appear that a slower heating rate would be detrimental to the densification of the conventionally fired body [Figure 6-6]. In fact, the figure suggested that a slower heating rate would actually result in alumina samples with higher density compared to those heated at a faster rate [John97].

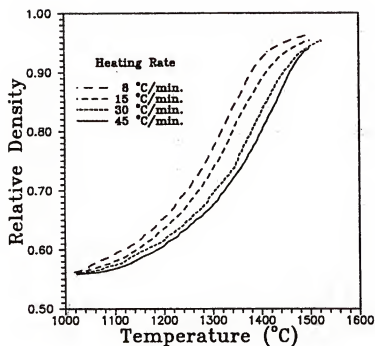


Figure 6-6. The Effect of Heating Rate on the Densification of Sumitomo AKP-50 Alumina [Su96].

Volumetric Heating

A more likely cause for the differences in densification produced in the Coors AD85 samples by the two firing techniques at identical firing temperatures was the volumetric heating phenomenon found in microwave heating. Volumetric heating of the alumina bodies could result in temperature gradients between the center and surface of the sample. Since the present study measured only the surface temperature of the body, the average body temperature could have been underestimated. This underestimate of average body temperature could have accounted for some, but not all, of the observed differences in densification.

Two studies provided some limits for the magnitude of this temperature variation throughout the sample [De90, Bran92]. Arrendum De [De90] measured the difference between the center and surface temperatures of microwave hybrid heated 8 gram and 25 gram 99.8% pure alumina pellets. The pellets had the same diameter as the pellets in the present study, and were microwave hybrid heated in same microwave oven as the one used in the present study. The differences between the center and surface temperatures of the pellets ranged from about $\sim 40\text{--}60^{\circ}\text{C}$ for the 8 gram sample, and was less than 10°C for the 25 gram sample [Figure 6-7].

Brandon [Bran92] measured the temperature difference between the center and the surface of larger 5 cm diameter, 6 cm thick pellets of 99.5% pure alumina samples and composites of 20wt% YSZ-alumina that were microwave hybrid heated at 2.45 GHz. After the firing temperature was reached, there remained a 20°C difference in temperature from the pure alumina samples, and a 30°C difference in temperature for the

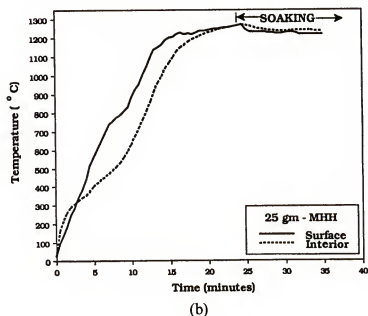
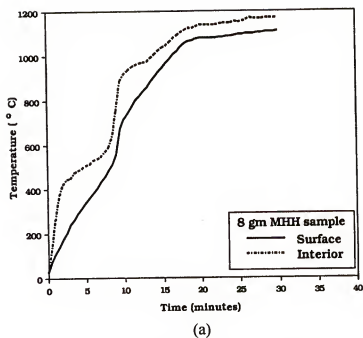


Figure 6-7. Temperature vs. Time Profile (Surface-Interior) for (a) 8 Gram and (b) 25 Gram Microwave Hybrid Heated (MHH) Alcoa A-16 Alumina Sample [De90].

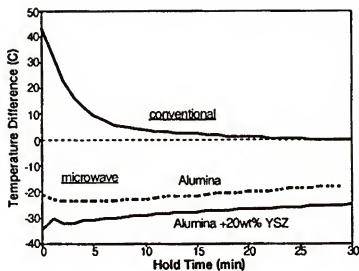


Figure 6-8. Comparative Volumetric Heating Data for Alumina and Alumina + 20wt% Yttria Stabilized Zirconia (YSZ) Specimens Held for 30 Minutes at 1500°C [Bran92].

composite samples [Figure 6-8]. It was important to note that the composite was more lossy than the pure alumina sample.

Based on the results of the De study, the difference in temperature between the center and surface of 99.8% pure alumina samples with similar masses as those used in the present study (12.7 gram and 15 gram) would be between 10 and 60°C. For the less pure and more lossy Coors AD85 alumina used in this study [Spot95], the results of Brandon suggested that the estimate of the temperature difference needed to be increased by about 10°C. It was therefore estimated that the difference in temperature between the center and surface of the microwave hybrid heated 12.7 gram and 15 gram Coors AD85 alumina samples could have been as high as 20 to 70°C.

Though a temperature gradient could have existed during the microwave hybrid heating of the samples, it did not account for all of densification enhancement found in this study. To account for the 75 to 200°C decrease in the firing temperatures required to achieve levels of densification in the Coors AD85 samples identical to those achieved using conventional firing, another mechanism, possibly non-thermal in nature, was at work.

Non-Thermal Effects

The Coors AD85 alumina samples had 25 volume percent of glass. Based on the [King59], 0.30 volume fraction of the shrinkage occurs by the rearrangement process (first stage of liquid-phase sintering) for bodies with 25 volume percent liquid phase. Since the starting density of the compacted samples was 58 to 59% theoretical density, the rearrangement process should account for all densification up to 88 to 89% of theoretical density. Because samples were sintered to a final density of 95% theoretical

density, this leaves only 6 to 7% of the remaining densification to occur by the solution-precipitation process (second stage) and solid-state skeletal sintering (third stage).

It was evident from comparisons of the densification curves for this alumina (Figure 5-1), that densification began at a lower temperature for microwave hybrid fired samples than for the conventionally fired samples. This decrease in the starting temperature for densification implied that the rearrangement process was being enhanced by microwave hybrid firing. The enhancement of the rearrangement process could have accounted for the densification enhancement found in this study due to the large dependence of this alumina on the rearrangement process for densification.

Possible causes for this enhanced densification by microwave hybrid firing were extracted from Equation 2-1. It was apparent from this equation that microwave hybrid firing either had to lower the apparent viscosity of the glass or increase the surface energy of the liquid relative found in conventional firing under identical conditions. Future experimental studies are needed to determine which of these variables is change by microwave firing.

Though the densification of the Coors AD85 alumina was dominated by the rearrangement process, it was possible that microwave hybrid firing also enhanced the small portion of densification that occurred by the solution-precipitation and solid-state sintering processes. For microwave hybrid firing to enhance the solution-precipitation process, it has to increase the diffusivity of the solid in the liquid and/or the energy of the liquid surface relative to conventional firing under identical conditions (Equation 2-2). Solid-state diffusion had to be enhanced if the solid-state sintering process was enhanced

by the presence of the microwave field. Several authors in the literature have suggested possible causes for the enhancement of diffusion in the microwave field (see Chapter 2).

Comparisons between Coors AD85 and Coors AD998 Alumina Powders

It has been shown that the difference between microwave and conventional densification of Coors AD998 powder was minimal at best [Figure 4-9]. On the other hand, there were large differences in the densification of Coors AD85 using the two processing techniques [Figures 5-1, and 5-2].

The cause for the increased enhancement of densification found in microwave hybrid heating of Coors AD85 alumina was rooted in the relative level of purity of the powder. Coors AD85 alumina powder was of lower purity than Coors AD998 alumina powder. With increased impurity in the powder came increased microwave absorption in powder relative to the more pure powder [Figure 6-9]. This increased microwave absorption resulted in increased microwave enhancement of densification found in the study.

The microwave enhancement of densification was more pronounced at the two lower firing temperatures (1200 and 1300°C) than at the highest firing temperature (1400°C). One study [Will95] found pronounced differences in grain morphology and densification behavior of alumina in the lower temperature region. At these lower sintering temperatures neck formation, surface area reduction and densification was much more advanced in microwave sintering as compared to conventional sintering at comparable temperatures.

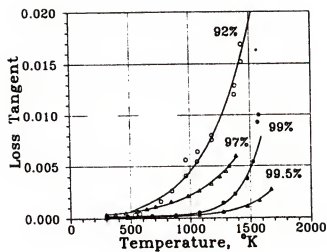


Figure 6-9. Dielectric Loss Tangent for Various Grades of Alumina [Spot95].

Because of this increased densification in the early stages of sintering, the microwave fired specimens approached maximum density at lower temperatures than conventionally fired samples. As Figure 2-9 showed, the densification rate greatly slows as the specimen approaches its maximum density. This decrease in the densification rate of the microwave fired specimens occurred at temperatures where the conventionally fired specimens were still rapidly densifying. Therefore, at these upper temperatures, the difference in densification between these two firing techniques narrowed.

It was interesting to note that there was little difference in densification rate of the microwave fired and conventionally fired Coors AD998 alumina samples [Figure 4-9].

Experiments on other alumina powders with similar purity have reported more noticeable differences in densification when comparing the two firing techniques [De90, Samu92, Chen92]. The major factor that was likely responsible for the diminished enhancement of densification found in the microwave fired Coors AD998 powder was the high level of agglomeration in the powder. Vodegel [Vode96] has found that high agglomeration substantially reduces the densification enhancement inherent to microwave firing. Unfortunately, it is not clear why this was the case.

Comparisons between this Study and Cited Data

Regardless of how accelerated densification occurred in microwave fired Coors AD85 alumina, it was evident from the results that the hardness and strength values for the microwave hybrid fired Coors AD85 samples (1400°C) were similar to the conventional fired samples (1500°C). This similarity in mechanical properties was not unreasonable since both sets of samples were compared at the same relative density and had qualitatively similar microstructures.

In addition to comparing favorably to the conventionally fired samples in this study, the mechanical properties of the microwave hybrid heated samples were also very similar to those cited on the Coors website as typical values for conventionally fired Coors AD85 alumina. Table 6-1 compares the hardness and fracture toughness of dense Coors AD85 samples fired in the current study to those cited on the website.

The fracture toughness values for the bars in this study were calculated using [Chan81b]

[Equ. 6-2]

$$K_c = \eta_r^v (E / H)^{1/8} (\sigma P^{1/3})^{3/4}$$

where,

- K_c = Fracture Toughness (MPa m^{1/2})
- η_r^v = constant (0.59+/-0.12; used 0.59 for calculation)
- E = Elastic Modulus for Coors AD85 (GPa)
- H = Vicker's Hardness for Coors AD85 (GPa)
- σ = Indented Four-Point Flexure Strength (MPa)
- P = Vicker's Hardness Indentation Load (N)

The elastic modulus for Coors AD85 alumina was determined from the Coors website to be 221 GPa, while 9.2 (conventional) to 9.6 (microwave) GPa (typical in this study) was used for the Vicker's hardness. The breaking stress at a 2 kg (19.6 N) and 10 kg (98N) indentation loads [Table 5-8] were used for the indented four-point flexure strength.

The website states that typical Coors AD85 bodies with densities of 3.4 g/cc have a Knoop hardness of 9.4 GPa when tested under a 1 kg load. This compares to a Vicker's hardness ranging from 9.0 to 10.7 GPa for the microwave hybrid heated samples in the

Table 6-1. Comparison of the Results of the Current Experiment to the Typical Mechanical Properties of Coors AD85 Alumina [Coor99]

<u>Sample</u>	Final Density (g/cc)	Hardness* ¹ (GPa)	Fracture Toughness (MPa m ^{1/2})
Coors AD85 (Typical)	3.4	9.4	3 to 4
Coors AD85 Microwave	3.45	9.0 to 10.1	2.9 * ²
Coors AD85 (conventional)	3.45	9.0 to 9.3	2.6 to 2.8 * ²

*¹ The hardness values cited as typical for Coors AD85 alumina are based on Knoop hardness measurements using a 1 Kg indentation load. A Vicker's hardness measurement technique with a 2 Kg indentation load was used to determine specimen hardness in the current study. *² Uncertainty in the calculations of fracture toughness for the conventional and microwave samples of this experiment was estimated to be +/-0.6 MPa m^{1/2}.

present study which were indented with a 2 kg load. The website suggests that Coors AD85 alumina typically has a fracture toughness that ranges from 3 to 4 MPa m^{1/2}. This compares to fracture toughnesses of 2.6 to 2.9 MPa m^{1/2}, for this study's microwave hybrid and conventionally fired bars, respectfully. When the uncertainties in the data and calculation were taken into account, the mechanical properties were similar to what is typical for Coors AD85 alumina.

Based on these comparisons, it appears that the Coors AD85 alumina samples that were produced in this study had reasonably similar final density and mechanical properties as what is typical for Coors AD85 alumina.

CHAPTER 7 SUMMARY AND CONCLUSIONS

Summary

It has been demonstrated that it is possible to microwave fire uniform batches of Coors AD85 alumina using 3200 W of 2.45 GHz microwave radiation and the susceptor/insulation system developed in this study. The susceptor developed in this study was a composite of silicon carbide particles in a matrix of alumina cement. It had the shape of a tube with a square cross-section, and was totally enclosed by a combination of alumina fiber board and alumina mat insulation.

The combination of microwave radiation and radiant heat from the susceptors successfully fired batches of ten to twelve 12.7 gram and 15 gram Coors AD85 alumina bars to ~95% relative density. The total processing time was about four times shorter for the microwave hybrid heated samples compared to the processing time used to fire identical samples in a conventional furnace. The reduction in processing time was partially due to the much faster heating rate obtained with microwave hybrid heating (~35°C/min for microwave firing vs. ~1.7°C/min for conventional firing).

In addition to requiring shorter total processing time, the firing temperature was from 75 to 200°C lower for the microwave hybrid heated samples to reach the same level of densification as the conventionally fired samples. This enhancement of densification was more pronounced at lower firing temperatures and gradually diminished as the firing temperature, and therefore sample density, was increased. The enhancement was likely

due to a combination of volumetric heating in the samples, which increased the average body temperature of samples relative to the measured surface temperature, and a non-thermal enhancement of the particle rearrangement stage (first stage) of liquid-phase sintering.

The extent of enhancement of densification in microwave hybrid firing seemed to be dependent on the level of microwave power that was directly interacting with the fired samples. The enhancements of densification for the 10 to 30 weight silicon carbide susceptors were very similar, owing to very small amounts of absorbed by the susceptor power (less than 14% of incident power was absorbed). The remaining 86+% of the microwave power was available for direct interaction with the Coors AD85 samples.

Firing with the 60 weight percent silicon carbide susceptor resulted in less enhancement of densification than was seen with the other three susceptors. This was likely due to the large microwave power absorption inherent to this susceptor. It was estimated from dielectric property calculations that the amount of incident power absorbed by this susceptor approached 35% at the higher firing temperatures used in this study. This left only 65% of the incident microwave power available for direct sample interaction.

Regardless of the susceptor used to fire samples, microwave hybrid heating was able produce samples with reasonably good mechanical properties. Comparisons at the same relative density (~95%) reveal that the hardness and indented strength were statistically similar for both microwave hybrid heated samples and conventionally processed samples. This level of densification was achieved at firing temperatures between 75 and 200°C lower than that required to densify the conventional samples.

The average Vicker's hardness for the microwave hybrid heated samples ranged from about 9.0 to 10.1 GPa. The indent strength of these samples was 185 and 106 MPa for Vicker's indents made with 2 and 10 Kg loads, respectfully. These hardness and strength values were reasonably comparable to values cited as typical for Coors AD85 alumina bodies having similar density.

The measured hardness values for both the microwave hybrid heated and conventionally fired were statistically uniform across the surface and through the thickness of the samples. This result showed that MHH can produce samples with uniform hardness.

Conclusions

Microwave hybrid heating is a viable alternative to conventional firing for the production of batches of small Coors AD85 alumina pieces. Microwave hybrid heating produces samples that are uniform and have mechanical properties equivalent to those of traditional firing techniques.

Advantages of this firing technique include firing temperatures that are from 75 to 200°C lower than those required for conventional sintering, shorter processing times afforded by faster ramp rates, and the ability to control densification (and possibly microstructure) by varying weight percent of absorbing phase in the susceptors. In addition, energy costs are lower for microwave hybrid heating compared to conventional firing techniques [Figure 1-1].

Decreasing the relative amount of microwave energy available for volumetric heating of the samples can reduce the rate of sample densification if the reduction of microwave energy is large enough. Microwave hybrid firing with 60 weight percent

silicon carbide susceptors having an additional 60 weight percent silicon carbide top absorbed enough of the incident microwave energy so that the densification rate of the Coors AD85 samples were slower than that of identical samples microwave hybrid-fired using susceptors with 30 weight percent or less silicon carbide. However, regardless of the susceptor chosen to microwave hybrid-fire the alumina Coors AD85 samples, sample densification occurred at a faster rate than that of the conventionally fired samples.

The limited number of studies on the more pure Coors AD998 alumina samples showed that its densification by microwave hybrid firing was very similar to that using conventional firing. This is in contrast to other studies throughout the literature which have found a relative densification enhancement for microwave firing of pure alumina specimens. A possible reason for the lack of densification enhancement for the microwave hybrid-fired Coors AD998 alumina is the high level of agglomeration in the powder.

A relative densification enhancement was provided by microwave hybrid firing of the less pure Coors AD85 powder. The firing temperature was from 75 to 200°C lower for the microwave hybrid heated Coors AD85 samples to reach the same level of densification as the conventionally fired samples. The enhancement was likely due to a combination of volumetric heating in the samples, which increased the average body temperature of samples relative to the measured surface temperature, and a non-thermal enhancement of the particle rearrangement stage (first stage) of liquid-phase sintering. The alumina (low purity) also absorbed more of the incident microwave energy relative to that absorbed by the Coors AD998 powder.

Based on the results of this study, microwave hybrid-firing appears to be a viable alternative to conventional firing for sintering of low purity alumina.

APPENDIX A BALLISTIC CONSIDERATIONS

Preface

This section is intended to provide some background information on an area that could benefit from microwave hybrid heating. Specifically, it provides insight into the mechanical and microstructural requirements for the production ceramic armor tile. This information, when coupled with a thorough understanding of microwave hybrid heating, provides a foundation for the development of this technique for firing of thin ceramic armor tile.

Ballistic Considerations

An armor ceramic can be defined as any ceramic functioning as the hard front face of a ballistic armor system. The primary roles of the armor ceramic are to damage or blunt the incoming projectile, and spread the impact load over a wide area so that the ductile backing is better able to absorb the residual kinetic energy.

Armor ceramics have found many applications since the First World War. Ceramic coatings have augmented tank armors [Viec91], small ceramic tile have been used in protective vests of air and ground personnel, while large ceramic monoliths have been applied to helicopter seats, armor panels, and military and civilian ground vehicles [Matc96]. They have been deployed against threats such as shell fragments, small rounds (both traditional and armor-piercing), long-rod penetrators, and shaped-charge

ammunition. The primary purpose of these applications has been to provide adequate protection at a reduced weight compared to traditional dual hardness metal armors.

The current light ceramic armor system employed against small armor-piercing (AP) threats consists of a thin, hard ceramic frontal plate bonded to a thin, metal or fiber-reinforced plastic backing and surrounded by a woven fiber spall cover. Schematics of current light armor systems and an AP round are provided in Figures A-1 and A-2. Each component is critical to the overall performance of the system. The ceramic face plays two important roles in the defeat of the projectile. It serves both to fragment and slow the incident projectile, as well as to distribute the projectile load to the ductile backing layer [Matc96]. The remaining projectile energy is then absorbed by the backing material.

Not all ceramic materials are meant to serve in armor applications. An effective ceramic must possess qualities such as low density, high hardness, tensile strength, and fracture toughness in order to defeat the incoming round. An overview of properties, relative cost, and processing techniques of four ceramics commonly used as frontal armor plate is provided in Table A-1. Of the four ceramics listed, boron carbide is considered to have the best ballistic performance, but the highest overall costs. There is a subsequent reduction in ballistic performance for the other three materials, with aluminum oxide having both the lowest cost and poorest performance.

The relative rank of ceramic armors is based on results from two tests used extensively throughout the literature: the V_{50} military standard test [MIL-STD-662E], and the Depth of Penetration (DOP) test [Wool91]. The V_{50} test involves shooting a series of shots onto a ceramic/backing material system and statistically determining the incident velocity at which 50% of the shots perforate the system. In DOP tests, the ceramic face

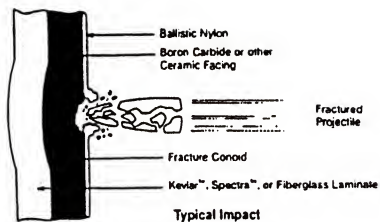


Figure A-1. Current Light Armor Systems [Matc96]

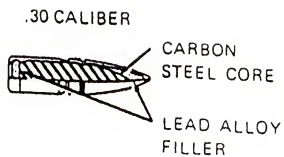


Figure A-2. Anatomy of an Armor-Piercing (AP) Round [Back78]

Table A-1. Overview of Four Commonly Used Ceramic Armor Materials [Viec87, Matc96]

Material	Boron Carbide	Silicon Carbide	Titanium Diboride	Alumina
Relative Cost	High	Moderate to high	Moderate to high	Moderate to Low
Processing Technique	Hot Press	Hot Press/Sinter	Hot Press/Sinter	Hot Press/Sinter
Hardness (GPa)	27.4	22.4	21.0	12.4
Young's Modulus ($\times 10^6$ psi)	65	59	71	54
Longitudinal Sonic velocity (m/s)	14000	12000	11000	11000
density (g/cm^3)	2.45	3.13	4.5	3.45-3.9
Flexure Strength ($\times 10^3$ psi)	67	50	-	40

plate is first mounted onto a semi-infinite metal substrate. The depth of penetration of the impacting projectile through the ceramic and into the substrate is recorded for comparison with other trials.

The results of the ballistic tests are dependent on several parameters such as the projectile impact velocity, target obliquity angle, ceramic type and thickness, backing material type and thickness [Wang96, Pesk96, Part93, Bles95, Hell94]. Impact performance can be improved by the proper adjustment of any one of these parameters.

An example of an armor system's dependence on the backing plate and ceramic thickness is provided in Figure A-3. The figure shows that there is an increase in the ballistic performance of ceramic armor systems with increases in the backing plate and ceramic thickness. It also shows that there is a transition (step) in the ballistic performance for a given ceramic thickness for backing plate thickness ranging from ~0.2 to 0.25 inches. This transition signifies a change in the failure mode of the armor system from tensile to plug failure.

Ballistic Failure Mechanics

The ballistic failure mechanics of armor ceramics are dependent on the striking velocity of the incident projectile. The response of ceramic armor systems can be divided into three major regimes [Figure A-4]. The low velocity regime ranges from impact velocities of up to 700 m/s, while the intermediate regime includes striking velocities from 700 m/s to 5000 m/s. The hypervelocity regime encompasses striking velocities above 5000 m/s.

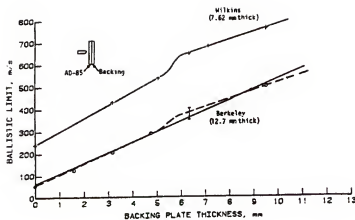


Figure A-3. Ballistic Limit of 6.35 mm AD-85 Alumina as a Function of 6061-T6 Backing Plate Thickness: Crosses: Data from Wilkins et. Al., (1969); Circles; Current Data. The Results Differ Due to Divergent Bullet Configurations. [Mays87, Wilk69].

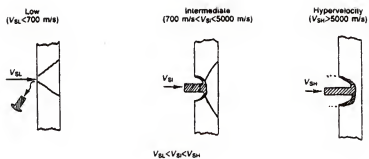


Figure A-4. Velocity Regimes of Ballistic Response (non-AP) [Viec91]

The striking velocities of military AP rounds fall in the low intermediate regime (700-1000 m/s), where penetration is governed by dynamic material properties and hydrodynamic flow [Figure A-5]. Penetration in this regime can be divided into four stages. Stage one includes the initial impact of the projectile on the ceramic and hydrodynamic flow. It is followed by the continuing flow of the penetrator into the ceramic with high speed jetting of ceramic debris (stage two). During stage three, the ceramic is fractured at the back and impact surfaces forming a tensile crack and fracture conoid, respectfully. The final stage in the penetration process involves widespread penetrator erosion and ceramic fracture.

The entire penetration process, from initial impact to widespread ceramic failure, is complete in a time scale on the order of tens of microseconds. A fundamental computational study [Wilk69] on 0.30 caliber AP striking a thin aluminum oxide/aluminum system suggests a timeline for the ballistic events. The timeline starts with the destruction of the projectile tip. This event occurs during the first 9 μ sec after impact. From 9 to 15 μ sec after impact, the projectile loses energy through erosion by the ceramic. After 15 μ sec, the erosion of the projectile stops and the crushed (comminuted) ceramic transmits the load to the backup plate across a smaller and smaller area, until the load is spread only over a projectile diameter.

The penetration phenomena in Figure A-4 and [Wilk69] are a direct result of the stress states induced in the ceramic upon initial projectile impact [Matc96, Figure A-6]. Impact by the projectile induces a compressive wave that propagates through the ceramic and projectile at speeds close to the materials' sonic velocity. When the compressive

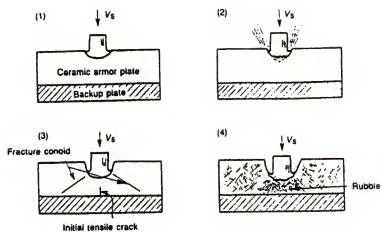


Figure A-5. Ballistic Response of Armor Ceramics in the Intermediate Velocity Regime [Viec91]

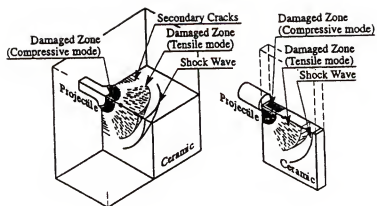


Figure A-6. Damage in Armor Ceramics during Ballistic Impact [Deno96]

waves reach free surfaces, they are reflected as tensile waves. The tensile waves cause the projectile to fragment, and the ceramic to fail in tension due to its inherently low tensile strength. The resulting failure in the ceramic occurs as a fracture conoid that radiates away from the impact point. This conoid distributes the impact load over an area much larger than a projectile diameter.

As the penetration process continues, the remaining projectile fragments continue to penetrate through a zone of fractured ceramic dubbed the comminuted zone [McGi95] (Figure A-7). The flow of ceramic particles opposite penetration erodes the projectile fragments and absorbs a significant amount of kinetic energy. Eventually, enough momentum is absorbed through erosion and deformation of the backup plate that all penetration is stopped. The round is then considered to be defeated.

In order to optimize the ceramic armor against the consequences of the impact stress state, the microstructure must be tailored to increase performance in each stage of impact failure. It is therefore important to review the stages of failure more closely, and identify the microstructural features that lead to improvements in ballistic performance.

Improving Armor Ceramics

The initial penetration of the projectile into the ceramic, the post-impact time of ceramic fracture, and the subsequent movement of the projectile through the fractured ceramic are all important to the overall performance of the armor. Through closer examination of these stages, insights are gained into the physical and mechanical properties governing the ballistic performance of ceramic armor. Knowledge about the

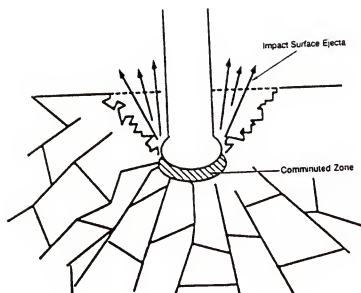


Figure A-7. Comminution in Ceramic Armor [McGi95]

nature and role of these properties can then be applied to the fabrication process and ceramics with tailored microstructures produced.

The initial resistance to projectile penetration is provided by the compressive strength or hardness of the ceramic [Skag90]. A study by Rosenberg and Yeshuran [Rose88] relates the ballistic efficiency of various ceramic armors to a combination of their strengths at high and low strain rates (effective strength) [Figure A-8]. When adjusted for density, the relationship is highly linear in nature, and the accepted ranking of ballistic performance [Table A-1] verified.

The role of the time of ceramic fracture is highlighted by the computational work of Wilkins [Wilk69]. Wilkins suggests that the important energy loss mechanism for a projectile that strikes a ceramic target is the loss of projectile mass. A 2 μ sec extension in the duration of projectile erosion by the ceramic results in a 10 percent increase in the ballistic performance. The total time of projectile erosion is directly related to the breakup of the ceramic. It is therefore important to maintain ceramic integrity for as long as possible. One way to increase this integrity time is to produce a new ceramic with improved tensile properties (while maintaining other properties), so as to delay the onset of the axial crack.

There are two possible routes to fabricate a more effective ceramic. One route is to produce a ceramic with improved tensile strength. The second way is to produce a ceramic that deforms plastically at low stress, but maintains the same ultimate tensile strength. In either case, there will be more strain before axial fracture occurs due to the tensile stress.

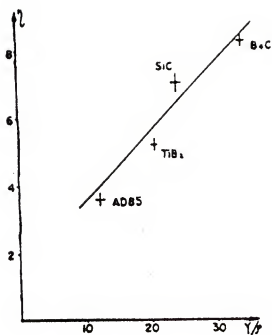


Figure A-8. Ballistic Efficiency vs. (Effective Strength/Density) [Rose88]

After formation of the axial crack, the projectile continues to penetrate into the ceramic and eventually a zone of comminution (rubble) is formed ahead of the penetrator [Figure A-7]. The comminuted zone [McGi95] is first comprised of coarse particles that have undergone intergranular fracture. As the process continues, the coarse ceramic particles are broken to the point of transgranular fracture. Nucleation sites for transgranular fracture included slip bands, inherent microvoids, and twins. The energy absorbed by both types of fracture is small relative to total energy absorbed by the penetration event. However, the shape and size of the resulting fragments greatly influence the energy absorbed in the flow of the material opposite the penetrator.

In the “comminution” stage, non-conventional properties such as the dynamic compressive failure energy and friction, flow, and abrasive properties are important. This stage tends to dominate the ballistic performance of thick, confined ceramics.

Microstructure and Ballistic Performance

Further review of the stages of ballistic failure has revealed that a number of mechanical and physical properties, both prior to and after fracture, are important to the overall ballistic performance of ceramic armor tile. These properties include the static and dynamic compressive strength, the static and dynamic tensile strength, the strain to failure, and the abrasive and flow characteristics of fractured ceramic particles. It is important to review the effect of microstructure on these properties, as well as any study directly linking a microstructural feature (i.e., grain size) to ballistic performance. This will establish a critical link between ballistic performance and the microstructure of the armor ceramic.

Two studies [Rais93, Stae95] on flyer plate impact on high-purity, vacuum hot-pressed alumina suggest that it is possible to improve ballistics-related properties by controlling microstructure. Specifically, an improvement in dynamic compressive properties of alumina is achieved by reducing the grain size, while a reduction in the amount of second phase improves the dynamic tensile strength of alumina. Reducing grain size makes the material less susceptible to inelastic deformation and sliding at triple junctions and grain boundaries by lowering average residual stresses at triple junctions. Decreasing the amount of glassy phases makes tensile damage less likely by increasing grain boundary strength.

In addition to grain size and purity, isometric grain structure, distribution of porosity, and special aligned grain boundaries also affect the strength of alumina. The presence of special aligned grain boundaries may be of great important to bulk material strength because they improve the bond strength of the material.

Unfortunately, flyer plate tests are not ballistic tests. They are designed only to create a condition of uniaxial strain in the ceramic. The stage of comminution and flow of fracture particles is also left unaddressed by flyer plate tests. Therefore, it is not possible to directly link these results to ballistic performance. What is needed is to review studies linking microstructure to ballistic performance, and then to design an experimental study to address unanswered questions.

There is a direct correlation between the dominant ballistic fracture mode and the relative ballistic performance. Ballistic performance is better for cases where transgranular fracture is the dominant fracture mode [Viec87, Rafa89]. Transgranular fracture may be indicative of plastic strain occurring during the failure event. Under the

right conditions, plastic strain could delay the onset of initial fracture, thereby increasing ballistic performance [Wilk69]. Unfortunately, the presence of the transgranular fracture mode could not be linked to grain size.

There have been a few studies in recent years that have attempted to link grain size and ballistic performance. A summary of these studies is presented in Table A-2. The studies tend to be very inconclusive, or at best marred by the presence of other uncontrolled microstructural characteristics (second phases, porosity).

Table A-2. Grain Size and Ballistic Performance

Study	Ceramic Material	Projectile	Range of Grain Sizes Studied (μm)	Correlation of Ballistic Performance with Grain Size
[Rafa89]	AlN(thin tile)	0.30 caliber AP projectile	1-12	No correlation
[Nels97]	SiC (thick tile)	long rod penetrator	1-5	Fine grain size showed better performance
[Nels97]	B ₄ C (thick tile)	long rod penetrator	expansive	No correlation
[Clin95]	SiC(thick tile)	tungsten heavy metal penetrator	1-15	No correlation
[Jame95]	Al ₂ O ₃ (thick tile)	¼ scale APFSDS penetrator	2-25	15 μm avg. grain size best

APPENDIX B
RAW DATA

Top surface hardness of 12.7 g Coors AD85 bar microwave hybrid fired using
10 wt% SiC susceptor

D1(μ m)	D2(μ m)	D1(mm)	D2(mm)	Davg(mm)	Hv(GPa)	Average Section Hv(GPa)	Section Uncertain (GPa)
69.4	65.1	0.0694	0.0651	0.0673	8.0	9.5	1.1
60.7	58.4	0.0607	0.0584	0.0596	10.3	Section 1	
63.2	62.4	0.0632	0.0624	0.0628	9.2		
59.5	61	0.0595	0.061	0.0603	10.0		
57.5	63.9	0.0575	0.0639	0.0607	9.9		
63.4	57.2	0.0634	0.0572	0.0603	10.0	10.7	1.5
56.8	56.6	0.0568	0.0566	0.0567	11.3	Section 2	
52.4	56.4	0.0524	0.0564	0.0544	12.3		
65.5	60.8	0.0655	0.0608	0.0632	9.1		
60.6	56.4	0.0606	0.0564	0.0585	10.6		
64.2	61.7	0.0642	0.0617	0.0630	9.2	10.5	1.6
63.7	61.7	0.0637	0.0617	0.0627	9.3	Section 3	
58.6	58.6	0.0586	0.0586	0.0586	10.6		
55.6	57.3	0.0556	0.0573	0.0565	11.4		
52.9	57.3	0.0529	0.0573	0.0551	12.0		
63.2	59.5	0.0632	0.0595	0.0614	9.7	9.1	0.9
66.9	64.7	0.0669	0.0647	0.0658	8.4	Section 4	
58.9	62.8	0.0589	0.0628	0.0609	9.8		
63.8	60.7	0.0638	0.0607	0.0623	9.4		
67	65.1	0.067	0.0651	0.0661	8.3		
54.2	60.6	0.0542	0.0606	0.0574	11.0	10.9	1.3
61.7	62.6	0.0617	0.0626	0.0622	9.4	Section 5	
57.3	57.6	0.0573	0.0576	0.0575	11.0		
55.8	53.2	0.0558	0.0532	0.0545	12.2		
57.1	59.8	0.0571	0.0598	0.0585	10.6		
Totals				Avg.	10.1 GPa		
				Stan Dev	1.2 GPa		
				95%Conf	0.5 GPa		
				Uncertain	0.5 GPa		

Top surface hardness of 12.7 g Coors AD85 bar microwave hybrid fired using
20 wt% SiC susceptor

D1(μ m)	D2(μ m)	D1(mm)	D2(mm)	Davg(mm)	Hv(GPa)	Average Section Hv(GPa)	Section Uncertain (GPa)
65.2	63.8	0.0652	0.0638	0.0645	8.7	9.2	0.5
66.1	64.4	0.0661	0.0644	0.06525	8.5	Section 1	
62.5	63.3	0.0625	0.0633	0.0629	9.2		
62.1	63.2	0.0621	0.0632	0.06265	9.3		
60.9	64.8	0.0609	0.0648	0.06285	9.2		
59.9	61.8	0.0599	0.0618	0.06085	9.8	8.9	0.9
62.8	62.6	0.0628	0.0626	0.0627	9.3	Section 2	
67.3	67.4	0.0673	0.0674	0.06735	8.0		
64.8	60.3	0.0648	0.0603	0.06255	9.3		
63.6	60.4	0.0636	0.0604	0.062	9.5		
66.5	66.1	0.0665	0.0661	0.0663	8.3	9.6	1.0
64.4	64.3	0.0644	0.0643	0.06435	8.8	Section 3	
63.7	61.7	0.0637	0.0617	0.0627	9.3		
60.9	61.8	0.0609	0.0618	0.06135	9.7		
59.2	60.2	0.0592	0.0602	0.0597	10.2		
59	60.4	0.059	0.0604	0.0597	10.2	9.3	1.9
55.3	60.5	0.0553	0.0605	0.0579	10.9	Section 4	
74.6	70.5	0.0746	0.0705	0.07255	6.9		
62	62.6	0.062	0.0626	0.0623	9.4		
60.4	62.2	0.0604	0.0622	0.0613	9.7		
60.4	61.6	0.0604	0.0616	0.061	9.8	7.4	0.5
61.3	63.2	0.0613	0.0632	0.06225	9.4	Section 5	
61	64	0.061	0.064	0.0625	9.3		
64.9	63.5	0.0649	0.0635	0.0642	8.8		
60.3	64.2	0.0603	0.0642	0.06225	9.4		
Totals				Avg.	9.2 GPa		
Bar F5				Stan Dev	0.8 GPa		
				95% conf	0.3 GPa		
				Uncertain	0.4 GPa		

Top surface hardness of 12.7 g Coors AD85 bar microwave hybrid fired using
30 wt% SiC susceptor

D1(μm)	D2(μm)	D1(mm)	D2(mm)	Davg(mm)	Hv(GPa)	Average Section Hv(GPa)	Section Uncertain (GPa)
57.4	62.2	0.0574	0.0622	0.0598	10.2	9.3	1
65.2	60.9	0.0652	0.0609	0.0631	9.2	Section 1	
67.1	67.1	0.0671	0.0671	0.0671	8.1		
61.6	60.9	0.0616	0.0609	0.0613	9.7		
60.7	63.3	0.0607	0.0633	0.0620	9.5		
62	61.4	0.062	0.0614	0.0617	9.6	9.8	0.5
61.1	58.2	0.0611	0.0582	0.0597	10.2	Section 2	
62.4	61.7	0.0624	0.0617	0.0621	9.4		
60	62	0.06	0.062	0.0610	9.8		
60.8	59.6	0.0608	0.0596	0.0602	10.0		
63	61.5	0.063	0.0615	0.0623	9.4	9.5	0.8
63.1	62.4	0.0631	0.0624	0.0628	9.2	Section 3	
64	61.3	0.064	0.0613	0.0627	9.3		
59.5	58.2	0.0595	0.0582	0.0589	10.5		
63.5	63.6	0.0635	0.0636	0.0636	9.0		
60.1	61.5	0.0601	0.0615	0.0608	9.8	9.9	0.7
61	59.1	0.061	0.0591	0.0601	10.1	Section 4	
62.3	63.5	0.0623	0.0635	0.0629	9.2		
60.5	60.9	0.0605	0.0609	0.0607	9.9		
56.7	59.8	0.0567	0.0598	0.0583	10.7		
62.9	59	0.0629	0.059	0.0610	9.8	9.6	0.5
63.5	62.4	0.0635	0.0624	0.0630	9.2	Section 5	
65	58.6	0.065	0.0586	0.0618	9.5		
60.8	58.6	0.0608	0.0586	0.0597	10.2		
61.5	62.4	0.0615	0.0624	0.0620	9.5		
Totals				Avg.	9.6 GPa		
				Stan Dev.	0.6 GPa		
				95% Conf	0.2 GPa		
				Uncertain	0.3 GPa		

Top surface hardness of 12.7 g Coors AD85 bar microwave hybrid fired using
60 wt% SiC susceptor with additional 60 wt% SiC top

						Average	Section	Section
						Section	Uncertain	
D1(μm)	D2(μm)	D1(mm)	D2(mm)	Davg(mm)	Hv(GPa)	Hv(GPa)	(GPa)	
62.8	65.6	0.0628	0.0656	0.0642	8.8	8.4	1.1	
70.7	74.7	0.0707	0.0747	0.0727	6.9	Section 1		
66.4	62.6	0.0664	0.0626	0.0645	8.7			
60.4	66.4	0.0604	0.0664	0.0634	9.1			
66.5	65.7	0.0665	0.0657	0.0661	8.3			
60.7	56.4	0.0607	0.0564	0.0586	10.6	9.6	1.1	
58.6	59.4	0.0586	0.0594	0.0590	10.5	Section 2		
65.1	62.8	0.0651	0.0628	0.0640	8.9			
62.9	65.5	0.0629	0.0655	0.0642	8.8			
62.2	63.1	0.0622	0.0631	0.0627	9.3			
59.5	59.5	0.0595	0.0595	0.0595	10.3	8.8	1.2	
61.6	63.8	0.0616	0.0638	0.0627	9.3	Section 3		
67.8	62.6	0.0678	0.0626	0.0652	8.6			
70.3	64.8	0.0703	0.0648	0.0676	8.0			
68.9	66.3	0.0689	0.0663	0.0676	8.0			
64.5	65.7	0.0645	0.0657	0.0651	8.6	9.0	0.8	
64	65.7	0.064	0.0657	0.0649	8.7	Section 4		
61.5	65.1	0.0615	0.0651	0.0633	9.1			
65	65.2	0.065	0.0652	0.0651	8.6			
61.8	59.1	0.0618	0.0591	0.0605	10.0			
61.3	61.4	0.0613	0.0614	0.0614	9.7	8.8	1	
59.8	63.4	0.0598	0.0634	0.0616	9.6	Section 5		
64.8	63.9	0.0648	0.0639	0.0644	8.8			
66	68.3	0.066	0.0683	0.0672	8.1			
65.7	69.5	0.0657	0.0695	0.0676	8.0			
Totals					Avg.	8.9 GPa		
					Stan Dev	0.5 GPa		
					95% conf	0.2 GPa		
					Uncertain	0.3 GPa		

Top surface hardness of conventionally fired 12.7 g Coors AD85 bar

D1(μm)	D2(μm)	D1(mm)	D2(mm)	Davg(mm)	Hv(GPa)	Average Section Hv(GPa)	Section Uncertain (GPa)
64.7	65.2	0.0647	0.0652	0.0650	8.6	9.3	0.6
62.8	58.4	0.0628	0.0584	0.0606	9.9	Section 1	
65.5	60.9	0.0655	0.0609	0.0632	9.1		
59.3	66.1	0.0593	0.0661	0.0627	9.3		
59.5	63.7	0.0595	0.0637	0.0616	9.6		
61.8	65.9	0.0618	0.0659	0.0639	8.9	8.7	0.9
64.9	68.2	0.0649	0.0682	0.0666	8.2	Section 2	
62.9	59.2	0.0629	0.0592	0.0611	9.8		
64.3	63.4	0.0643	0.0634	0.0639	8.9		
68.8	67	0.0688	0.067	0.0679	7.9		
60.3	63.7	0.0603	0.0637	0.0620	9.5	8.3	1.7
65	67.8	0.065	0.0678	0.0664	8.3	Section 3	
73.1	75	0.0731	0.075	0.0741	6.6		
70.1	69.1	0.0701	0.0691	0.0696	7.5		
61.1	60.9	0.0611	0.0609	0.0610	9.8		
61.7	60.7	0.0617	0.0607	0.0612	9.7	9.3	0.6
61.2	61.5	0.0612	0.0615	0.0614	9.7	Section 4	
61.9	65.2	0.0619	0.0652	0.0636	9.0		
62.7	62.3	0.0627	0.0623	0.0625	9.3		
66.2	64	0.0662	0.064	0.0651	8.6		
61.9	63.5	0.0619	0.0635	0.0627	9.3	9.3	0.7
65	64.9	0.065	0.0649	0.0650	8.6	Section 5	
64.6	61.9	0.0646	0.0619	0.0633	9.1		
63.9	61.4	0.0639	0.0614	0.0627	9.3		
60.9	59.5	0.0609	0.0595	0.0602	10.0		
Totals				Avg.	9.0 GPa		
				Stan Dev	0.8 GPa		
				95% conf	0.3 GPa		
				Uncertain	0.6 GPa		

Top surface hardness of 15 g Coors AD85 bar microwave hybrid fired using
20 wt% SiC susceptor

						Average	Section	Section Uncertain
D1(μm)	D2(μm)	D1(mm)	D2(mm)	Davg(mm)	Hv(GPa)	Hv(GPa)	(GPa)	
62	65.8	0.062	0.0658	0.0639	8.9	7.9		1.3
76.7	73.5	0.0767	0.0735	0.0751	6.5	Section 1		
66.9	71.1	0.0669	0.0711	0.0690	7.6			
61.5	65.7	0.0615	0.0657	0.0636	9.0			
70.1	67.9	0.0701	0.0679	0.0690	7.6			
62.5	56.1	0.0625	0.0561	0.0593	10.3	10.1		0.7
58.7	58	0.0587	0.058	0.0584	10.7	Section 2		
61.5	59.5	0.0615	0.0595	0.0605	9.9			
62	58.5	0.062	0.0585	0.0603	10.0			
64.1	61.3	0.0641	0.0613	0.0627	9.3			
60	60.7	0.06	0.0607	0.0604	10.0	9.7		0.4
64	60.7	0.064	0.0607	0.0624	9.4	Section 3		
60.3	61.2	0.0603	0.0612	0.0608	9.9			
65.3	58.6	0.0653	0.0586	0.0620	9.5			
61.6	59.1	0.0616	0.0591	0.0604	10.0			
63.6	70.6	0.0636	0.0706	0.0671	8.1	10.4		2
60.4	62.2	0.0604	0.0622	0.0613	9.7	Section 4		
54.1	55.1	0.0541	0.0551	0.0546	12.2			
58.1	55	0.0581	0.055	0.0566	11.4			
58.8	59.4	0.0588	0.0594	0.0591	10.4			
66	66.1	0.066	0.0661	0.0661	8.3	8.3		0.7
65.1	66.7	0.0651	0.0667	0.0659	8.4	Section 5		
62.3	67.7	0.0623	0.0677	0.0650	8.6			
68.2	60.4	0.0682	0.0604	0.0643	8.8			
71.1	69.1	0.0711	0.0691	0.0701	7.4			
Totals				Avg.	9.3 GPa			
				Stan Dev	1.3 GPa			
				95% conf	0.5 GPa			
				Uncertain	0.6 GPa			

Top surface hardness of conventionally fired 15 g Coors AD85 bar

D1(μ m)	D2(μ m)	D1(mm)	D2(mm)	Davg(mm)	Hv(GPa)	Average	Section	Section Uncertainty (GPa)
						Hv(GPa)		
54.7	63.3	0.0547	0.0633	0.0590	10.5	10.4	Section 1	1.6
59.8	63.2	0.0598	0.0632	0.0615	9.6			
57.8	56.8	0.0578	0.0568	0.0573	11.1			
54.7	54.6	0.0547	0.0546	0.0547	12.2			
64.9	63.4	0.0649	0.0634	0.0642	8.8			
61.8	70.9	0.0618	0.0709	0.0664	8.3	9.6		1.8
61.7	64.5	0.0617	0.0645	0.0631	9.1		Section 2	
67.8	63.8	0.0678	0.0638	0.0658	8.4			
55.7	56.7	0.0557	0.0567	0.0562	11.5			
57.2	60.7	0.0572	0.0607	0.0590	10.5			
63.4	67.6	0.0634	0.0676	0.0655	8.5	9.1		0.9
60.6	65.8	0.0606	0.0658	0.0632	9.1		Section 3	
61.1	60	0.0611	0.06	0.0606	9.9			
65.9	66.3	0.0659	0.0663	0.0661	8.3			
59.5	62.9	0.0595	0.0629	0.0612	9.7			
64.5	63.9	0.0645	0.0639	0.0642	8.8	8.3		1.2
70.4	62.6	0.0704	0.0626	0.0665	8.2		Section 4	
61.3	62.1	0.0613	0.0621	0.0617	9.6			
67.9	68.1	0.0679	0.0681	0.0680	7.9			
64	78.8	0.064	0.0788	0.0714	7.1			
61.3	68.7	0.0613	0.0687	0.0650	8.6	9.3		1.1
59.9	61.5	0.0599	0.0615	0.0607	9.9		Section 5	
59.9	60.2	0.0599	0.0602	0.0601	10.1			
58.7	63.6	0.0587	0.0636	0.0612	9.7			
68.5	64.4	0.0685	0.0644	0.0665	8.2			
72.8	64.5	0.0728	0.0645	0.0687	7.7			
				Totals	Avg.	9.3 GPa		
					Stan Dev	1.2 GPa		
					95% conf	0.5 GPa		
					Uncertainty	0.6 GPa		

Interior Hardness measurements on 12.7 g Coors AD85 bar microwave hybrid fired using
10 wt% SiC susceptor

<u>D1(μm)</u>	<u>D2(μm)</u>	<u>D1(mm)</u>	<u>D2(mm)</u>	<u>Davg(mm)</u>	<u>Hv(Gpa)</u>	Average Section <u>Hv(GPa)</u>	Section Uncertain <u>(GPa)</u>
58.6	58.8	0.0586	0.0588	0.0587	10.6	7.9	2.5
66.2	64.8	0.0662	0.0648	0.0655	8.5	Interior	
70.9	71	0.0709	0.071	0.0710	7.2		
64.2	67.2	0.0642	0.0672	0.0657	8.4		
82.3	87.9	0.0823	0.0879	0.0851	5.0		
70.1	68.1	0.0701	0.0681	0.0691	7.6	7.6	1.6
61.3	60.6	0.0613	0.0606	0.0610	9.8	Near	
71.5	73.1	0.0715	0.0731	0.0723	7.0	Surface	
76.7	70.1	0.0767	0.0701	0.0734	6.8		
72.2	73.3	0.0722	0.0733	0.0728	6.9		

Interior Hardness measurements on 12.7 g Coors AD85 bar microwave hybrid fired using
20 wt% SiC susceptor

<u>D1(μm)</u>	<u>D2(μm)</u>	<u>D1(mm)</u>	<u>D2(mm)</u>	<u>Davg(mm)</u>	<u>Hv(Gpa)</u>	Average Section <u>Hv(GPa)</u>	Section Uncertain <u>(GPa)</u>
64.1	63.9	0.0641	0.0639	0.0640	8.9	9.4	1.2
66.4	64.7	0.0664	0.0647	0.0656	8.5	Interior	
58.5	61.2	0.0585	0.0612	0.0599	10.2		
60.1	56.9	0.0601	0.0569	0.0585	10.6		
64.7	63.9	0.0647	0.0639	0.0643	8.8		
63.4	63	0.0634	0.063	0.0632	9.1	9.2	1.1
60.1	61.4	0.0601	0.0614	0.0608	9.9	Near	
61.4	64.8	0.0614	0.0648	0.0631	9.1	Surface	
68.7	67.5	0.0687	0.0675	0.0681	7.8		
57.9	62.4	0.0579	0.0624	0.0602	10.1		

Interior Hardness measurements on 12.7 g Coors AD85 bar microwave hybrid fired using
30 wt% SiC susceptor

<u>D1(μm)</u>	<u>D2(μm)</u>	<u>D1(mm)</u>	<u>D2(mm)</u>	<u>Davg(mm)</u>	<u>Hv(Gpa)</u>	Average Section <u>Hv(GPa)</u>	Section Uncertain <u>(GPa)</u>
62.7	64.3	0.0627	0.0643	0.0635	9.0	9.4	1
57.6	59.2	0.0576	0.0592	0.0584	10.7	Interior	
62.5	67.4	0.0625	0.0674	0.0650	8.6		
64.9	61.4	0.0649	0.0614	0.0632	9.1		
61.8	61	0.0618	0.061	0.0614	9.7		
55.2	58.9	0.0552	0.0589	0.0571	11.2	10.2	1.2
64	62.1	0.064	0.0621	0.0631	9.2	Near	
60.5	62.7	0.0605	0.0627	0.0616	9.6	Surface	
59.5	54.7	0.0595	0.0547	0.0571	11.2		
61.8	60.4	0.0618	0.0604	0.0611	9.7		

Interior Hardness measurements on 15 g Coors AD85 bar microwave hybrid fired using 20 wt% SiC susceptor

<u>D1(μm)</u>	<u>D2(μm)</u>	<u>D1(mm)</u>	<u>D2(mm)</u>	<u>Davg(mm)</u>	<u>Hv(GPa)</u>	Average Section <u>Hv(GPa)</u>	Section Uncertain <u>(GPa)</u>
60	60.8	0.06	0.0608	0.0604	10.0	8.2	1.4
72.5	68.1	0.0725	0.0681	0.0703	7.4	Interior	
66.5	62.9	0.0665	0.0629	0.0647	8.7		
70.2	68.9	0.0702	0.0689	0.0696	7.5		
70.7	69.8	0.0707	0.0698	0.0703	7.4		
67.3	66.1	0.0673	0.0661	0.0667	8.2	8.5	1.2
66.5	64.9	0.0665	0.0649	0.0657	8.4	Near	
59	60.6	0.059	0.0606	0.0598	10.2	Surface	
67.1	70	0.0671	0.07	0.0686	7.7		
67.6	65.6	0.0676	0.0656	0.0666	8.2		

Interior hardness measurements on conventionally fired 15 g Coors AD85 bar

<u>D1(μm)</u>	<u>D2(μm)</u>	<u>D1(mm)</u>	<u>D2(mm)</u>	<u>Davg(mm)</u>	<u>Hv(GPa)</u>	Average Section <u>Hv(GPa)</u>	Section Uncertain <u>(GPa)</u>
62	62	0.062	0.062	0.0620	9.5	9.6	2.5
54.2	54.2	0.0542	0.0542	0.0542	12.4	Interior	
58.6	58.6	0.0586	0.0586	0.0586	10.6		
66	66	0.066	0.066	0.0660	8.4		
70.6	70.6	0.0706	0.0706	0.0706	7.3		
70.5	70.5	0.0705	0.0705	0.0705	7.3	8.7	1.6
63.7	63.7	0.0637	0.0637	0.0637	9.0	Near	
67.2	67.2	0.0672	0.0672	0.0672	8.1	Surface	
65.8	65.8	0.0658	0.0658	0.0658	8.4		
58.4	58.4	0.0584	0.0584	0.0584	10.7		

Interior Hardness measurements on 12.7 g Coors AD85 bar microwave hybrid fired using 60 wt% SiC susceptor with additional 60 wt% SiC top

<u>D1(μm)</u>	<u>D2(μm)</u>	<u>D1(mm)</u>	<u>D2(mm)</u>	<u>Davg(mm)</u>	<u>Hv(GPa)</u>	<u>Average Section Hv(GPa)</u>	<u>Section Uncertain (GPa)</u>
64.8	64	0.0648	0.064	0.0644	8.8	9.2	0.6
60.5	64.8	0.0605	0.0648	0.0627	9.3	Interior	
62.1	58.7	0.0621	0.0587	0.0604	10.0		
61.8	65.3	0.0618	0.0653	0.0636	9.0		
63.5	64	0.0635	0.064	0.0638	9.0		
56.6	61.3	0.0566	0.0613	0.0590	10.5	9.0	1.7
66.5	66.3	0.0665	0.0663	0.0664	8.3	Near	
71	71.3	0.071	0.0713	0.0712	7.2	Surface	
64.9	62.9	0.0649	0.0629	0.0639	8.9		
61	58.6	0.061	0.0586	0.0598	10.2		

Interior hardness measurements on conventionally fired 12.7 g Coors AD85 bar

<u>D1(μm)</u>	<u>D2(μm)</u>	<u>D1(mm)</u>	<u>D2(mm)</u>	<u>Davg(mm)</u>	<u>Hv(GPa)</u>	<u>Average Section Hv(GPa)</u>	<u>Section Uncertain (GPa)</u>
62.6	60.9	0.0626	0.0609	0.0618	9.5	9.4	0.3
62.3	64	0.0623	0.064	0.0632	9.1	Interior	
60	64.5	0.06	0.0645	0.0623	9.4		
62	62	0.062	0.062	0.0620	9.5		
62	62	0.062	0.062	0.0620	9.5		
83.2	82.3	0.0832	0.0823	0.0828	5.3	8.3	2.6
69.2	66.9	0.0692	0.0669	0.0681	7.9	Near	
66.6	64.9	0.0666	0.0649	0.0658	8.4	Surface	
54.7	59.6	0.0547	0.0596	0.0572	11.1		
65.1	64.6	0.0651	0.0646	0.0649	8.7		

Raw Data for Indented 4-Point Flexure Testing on Microwave Hybrid Fired
12.7 g Coors AD85 Bars using 20 wt% SiC Susceptor

Sample #	Indentation		Thick (in)	Width (cm)	Width (in.)	Span (cm)
	Load (N)	Thick (cm)				
2kgT1	19.6	0.73	0.29	0.8	0.31	4
2kgB2	19.6	0.74	0.29	0.79	0.31	4
2kgB4	19.6	0.73	0.29	0.81	0.32	4
2kgT4	19.6	0.73	0.29	0.8	0.31	4
2kgT5	19.6	0.75	0.30	0.79	0.31	4
10kgT1	98	0.73	0.29	0.8	0.31	4
10kgB2	98	0.74	0.29	0.8	0.31	4
10kgT3	98	0.74	0.29	0.79	0.31	4
10kgT4	98	0.74	0.29	0.79	0.31	4
10kgB5	98	0.74	0.29	0.81	0.32	4

Span (in)	Breaking Load (lb)	Breaking Stress (psi)	Breaking Stress (GPa)	Average	Uncertainty (Gpa)
				Breaking Stress (GPa)	
1.57	600	27240	187.809017	185	15
1.57	540	24160	166.572815		
1.57	620	27800	191.6734		
1.57	630	28602	197.199468		
1.57	610	26569	183.18129		
1.57	330	14982	103.294959	107	9
1.57	390	17231	118.798806		
1.57	320	14317	98.7098165		
1.57	340	15212	104.87918		
1.57	360	15709	108.306604		

Raw Data for Indented 4-Point Flexure Testing on Microwave Hybrid Fired
12.7 g Coors AD85 Bars using 60 wt% SiC Susceptor with additional 60 wt% SiC top

Sample #	Indentation		Thick (in)	Width (cm)	Width (in.)	Span (cm)
	Load (N)	Thick (cm)				
2kgT1	19.6	0.75	0.30	0.79	0.31	4
2kgB2	19.6	0.73	0.29	0.8	0.31	4
2kgB4	19.6	0.76	0.30	0.79	0.31	4
2kgT4	19.6	0.73	0.29	0.79	0.31	4
10kgT1	98	0.74	0.29	0.8	0.31	4
10kgT2	98	0.74	0.29	0.8	0.31	4
10kgB3	98	0.76	0.30	0.79	0.31	4
10kgT5	98	0.74	0.29	0.8	0.31	4

Span (in)	Breaking Load (lb)	Breaking Stress (psi)	Breaking Stress (GPa)	Average	
				Breaking Stress (GPa)	Uncertainty (Gpa)
1.57	650	28311	195	185	18
1.57	590	26786	185		
1.57	580	24602	170		
1.57	600	27585	190		
1.57	330	14580	101	103	10
1.57	330	14580	101		
1.57	350	14846	102		
1.57	360	15905	110		

Raw Data for Indented 4-Point Flexure Testing on Conventionally Fired
12.7 g Coors AD85 bars

Sample #	Indentation		Thick (in)	Width (cm)	Width (in.)	Span (cm)
	Load (N)	Thick (cm)				
2kgB2	19.6	0.72	0.28	0.8	0.31	4
2kgB3	19.6	0.76	0.30	0.78	0.31	4
2kgT4	19.6	0.72	0.28	0.8	0.31	4
2kgT1	19.6	0.72	0.28	0.8	0.31	4
10kgT1	98	0.73	0.29	0.8	0.31	4
10kgB2	98	0.73	0.29	0.8	0.31	4
10kgT3	98	0.75	0.30	0.8	0.31	4
10kgB4	98	0.74	0.29	0.8	0.31	4
10kgT5	98	0.73	0.29	0.8	0.31	4

Span (in)	Breaking Load (lb)	Breaking Stress (psi)	Breaking Stress (GPa)	Average	
				Breaking Stress (GPa)	Uncertainty (Gpa)
1.57	600	28002	193	176	21
1.57	600	25776	178		
1.57	500	23335	161		
1.57	540	25202	174		
1.57	310	14074	97	90	9
1.57	300	13620	94		
1.57	270	11613	80		
1.57	310	13696	94		
1.57	270	12258	85		

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BIOGRAPHICAL SKETCH

Jerry Mark Moore was born in Port Arthur, Texas, on January 6, 1970, to Mr. and Mrs. Jerry Marshall Moore. He was raised in Groves, a smaller town adjacent to Port Arthur. Through training received at his home church (Procter Baptist Church), and through the example set by his parents, he accepted Jesus as his savior at the age of eight. From that point forward, through the many opportunities life provides, his relationship with God continues to grow.

After high school at Port Neches-Groves High School, Mark began studies in mechanical engineering at Texas Agricultural and Mechanical University. While attending college, he enjoyed the many traditions, camaraderie, and friendliness that make Texas A&M University such a special place. He graduated in December of 1992 with a bachelor's degree in mechanical engineering.

In January of 1993, Mark began advanced studies in mechanical engineering at Clemson University in Clemson, South Carolina, under the direction of Dr. Richard S. Figliola. His research was in the area of cooling of electronic packages. Much was learned from the challenges provided by the master's thesis, which was completed in time for graduation in December of 1995.

From August of 1994 to December of 1999, Mark has worked on a doctorate in materials science and engineering at the University of Florida under the direction of David E. Clark. During his time at the University of Florida, he has had the opportunity

to work on several research projects, including microwave processing of sol-gels, glass corrosion, temperature measurement in a microwave field, and firing of ceramic materials. He has also had the opportunity to make many friends through membership at First Baptist-Gainesville, and participation in the Baptist Student Union, Fellowship of Christian Athletes, Dr. Clark's research group, and the Department of Materials Science and Engineering.

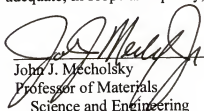
He looks forward to the next place that God will lead him, after his graduation in December 1999.

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.



David E. Clark, Chair
Professor of Materials
Science and Engineering

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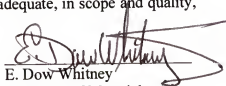
John J. Mecholsky
Professor of Materials
Science and Engineering

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Robert T. DeHoff
Professor of Materials
Science and Engineering

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E. Dow Whitney
Professor of Materials
Science and Engineering

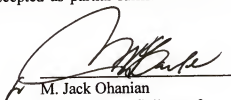
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Professor of Aerospace
Engineering, Mechanics
and Engineering Science

This dissertation was submitted to the Graduate Faculty of the College of Engineering and to the Graduate School and was accepted as partial fulfillment of the requirements for the degree of Doctor of Philosophy

December 1999

A handwritten signature in black ink, appearing to read 'M. Jack Ohanian', is written over a horizontal line.

M. Jack Ohanian
Interim Dean, College of
Engineering

Winfred M. Phillips
Dean, Graduate School